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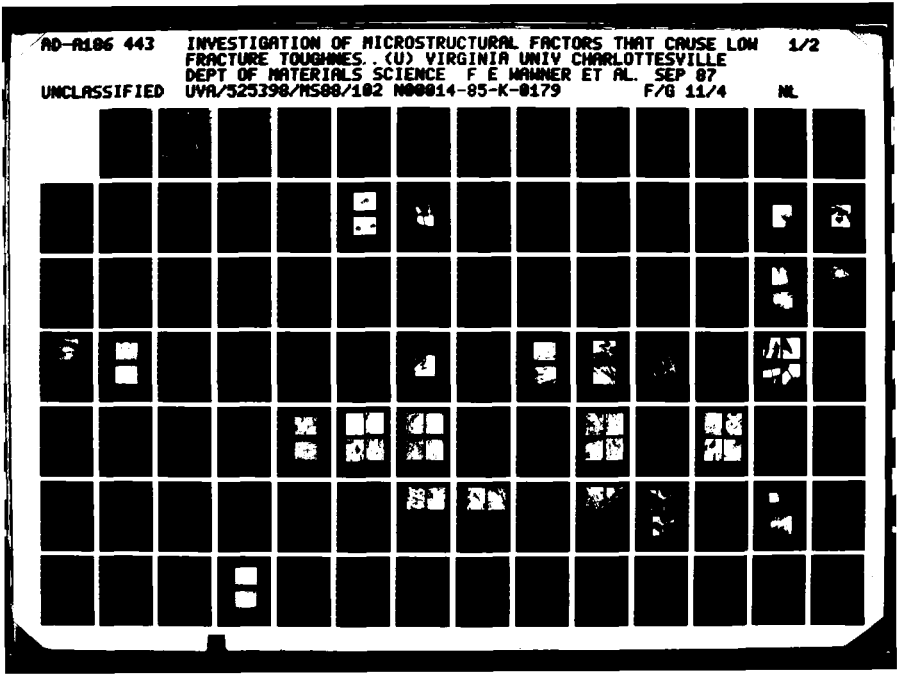

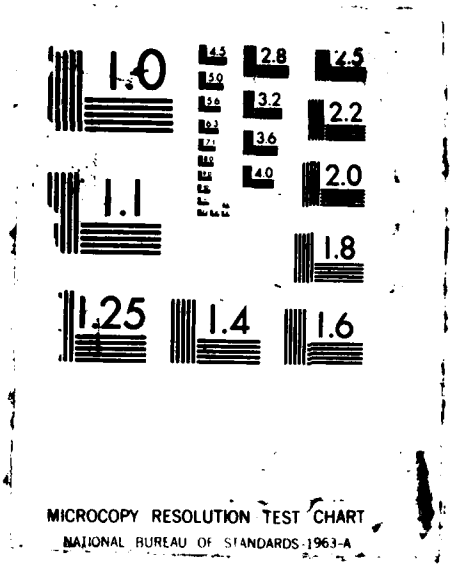
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Annual Report
Grant No. N00014-85-K0179
September 1, 1986 - September 30, 1987

INVESTIGATION OF MICROSTRUCTURAL FACTORS THAT CAUSE LOW FRACTURE TOUGHNESS IN SILICON CARBIDE WHISKER/AL ALLOY COMPOSITES

Submitted to:

Office of Naval Research
800 N. Quincy Street
Arlington, VA 22217-5000

Attention: Dr. Steven Fishman
Non-Metallic Materials
Code 1131N

Submitted by:

F. E. Wawner
Research Professor

C. R. Harris
Graduate Research Assistant

Report No. UVA/525398/MS88/102
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SCHOOL OF ENGINEERING AND APPLIED SCIENCE

DEPARTMENT OF MATERIALS SCIENCE

UNIVERSITY OF VIRGINIA
CHARLOTTESVILLE, VIRGINIA 22901

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19 ABSTRACT (*Continue on reverse if necessary and identify by block number*)

This investigation deals with factors influencing the fracture toughness in silicon carbide whisker - aluminum (SiC_w-Al) alloy composites. There is an enormous amount of variables which can, independently or in combination, yield different properties in these materials. Chemical composition, manufacturing techniques and processing, along with the material's history, must all be considered for a complete understanding of the material's behavior.

The approach employed here is to evaluate strength parameters in order to gauge the effectiveness of one variable, individually altered, while all other variables remain constant. For example, ultimate tensile strength was one parameter measured before and after a certain heat treatment. Any observed changes in strength were then investigated on a microstructural level, using electron microscopy and x-ray techniques. Ideally, an explanation for the phenomenon can be found and a model proposed to explain or predict the behavior.

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Originally, chemical variables, such as alloying additions to the Al matrix were investigated, by characterization of the composites before and after compositional changes were made. It was found that elimination of large matrix precipitates resulted in improved mechanical properties.

Later, thermal and mechanical processes were chosen as variables to be investigated. Again, any observed changes were examined on a microstructural level. The integrity of the fiber-matrix interface was found to be an important variable in mechanical processing. Work hardening is an effective strengthening mechanism in aluminum alloys; in composites, however, it can degrade strength if interfacial damage is incurred during the deformation process. Matrix dislocation strengthening, induced by rolling, was seen to be offset by damage to the interface. However, indications were also found, that further processing (heat treatment) can, in some instances, repair some of the interfacial damage.

Strength was found to depend on annealing temperature. In 2124 composites, excessive grain boundary precipitation of manganese-containing particles takes place at a temperature of 270°C. This phenomenon was determined to be very detrimental to these materials. A drastic reduction in tensile strength was seen for composites which were exposed at that temperature, for short periods of time. Thermal studies also revealed that the presence of silicon carbide particles in Al composites alters the solidus temperature and melting characteristics of the matrix material.

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LIST OF SYMBOLS

cm - Centimeter

D - Fiber Diameter

DSC - Differential Scanning Calorimetry

EDS - Energy Dispersive Spectroscopy

GPa - Gigapascal

Ksi - Kips per Square Inch

L_c - Critical Fiber Length

L/D - Length-to-Diameter Ratio, or Aspect Ratio

mm - Millimeter

MPa - Megapascal

psi - Pounds per Square Inch

S_f - Maximum Fiber Tensile Strength

SEM - Scanning Electron Microscopy (or Microscope)

SiC - Silicon Carbide

SiC_w - Silicon Carbide Whisker(s)

Silag - Tradename for Silicon Carbide Whiskers Produced by ARCO Metals

t_m - Shear Yield Stress of Matrix

TEM - Transmission Electron Microscopy (or Microscope)

v/o - Volume %

INTRODUCTION

Correlation of microstructural features with mechanical properties and processing techniques, has been carried out to identify factors that influence failure modes in aluminum-silicon carbide composites. The objective of this study is to investigate material variables, mainly thermomechanical processing, which affect tensile strength and elongation in SiC_w-Al matrix composites. Correlating mechanical properties with microstructure, identifying failure modes, and evaluating thermomechanical processes for improvement of composites was the approach taken in this study.

Lighter, stronger, structural materials have been sought after for many years. Finding materials for new applications, and also replacements for presently used materials, is an area of active research. Many of the conventional metals and their alloying components (i.e. additives such as Ti, Cr, Co) are imported from potentially unstable nations and have been placed on a critical materials list. For this reason, replacement of these materials may become a necessity in the very near future. Metal matrix composites are being considered to fulfill this role.

The problem of high cost which has inhibited the use of metal matrix composite materials for many years is slowly being alleviated. Improved manufacturing techniques, higher

volume usage, and longer material life have led to increased consideration for their use in present and future design concepts.

One such materials system which has moved rapidly from its laboratory inception to near commercialization is a silicon carbide whisker reinforced aluminum composite, known as Silag/Al (Silag is a tradename for SiC whiskers produced by ARCO Metals.) This material is typically composed of 20 volume percent SiC whiskers in various aluminum matrices. It has demonstrated strength values that equal or exceed most Al alloys, and possesses a Young's modulus that is 75% higher (18×10^6 psi [124 GPa]). These properties are quite similar to those obtained for many titanium alloys. However, aluminum composites are lighter in weight, hence their specific properties are even more impressive. These properties alone are sufficient justification to consider further work with this materials system, but additional advantages of being able to use conventional forming methods such as extrusion, forging, rolling, etc. make the materials attractive from an economic and a convenience standpoint.

The development of silicon carbide whisker reinforced composite material has followed a logical progression, in that baseline mechanical property data has been established. Additionally, a limited amount of microstructural characterization has been performed. As with many materials, success comes quickly in the early stages of development, but

then more difficult problems are encountered at a later date. These difficulties are overcome only by obtaining a complete understanding the system. Most frequently the limitations are related to the microstructure, hence correlation of specimen history and properties with microstructural observations often leads to a better understanding of the material.

Fracture toughness and elongation to failure of many composites is considerably lower than desirable [1]. Investigations need to be made on the microstructural level and mechanisms proposed for the actual failure processes. Using this information, alternatives can be developed to minimize the deleterious factors leading to low toughness and elongation in the discontinuous SiC whisker composite system. It is to this problem that the present study is addressed.

It has long been accepted that composite materials made using long, continuous, high strength reinforcement, achieve overall improved strength properties. This is accomplished by transferring applied loads through the matrix to the reinforcement, which can then accommodate the higher stress. However, in the case of relatively short (10 micron) SiC whiskers, it is questionable whether or not the whiskers are long enough to support a load. Evidence will later be presented to show that small SiC whiskers can indeed carry a load transferred through the matrix, and that this phenomenon must be considered the major strengthening mechanism in SiC_w-Al composites.

Failure modes were investigated. Cracks in composites can theoretically initiate at many different sites within the material. Evidence will be given identifying the most detrimental crack initiation sites. Also, several compositional and processing changes were investigated in an attempt to alleviate these problems. Both property enhancing treatments, and also some previously unrecognized harmful processes and treatments will be identified. Recommendations to avoid or alleviate problems were evaluated and will be presented.

BACKGROUND

A great deal of research in the past twenty years has been done in the field of reinforcing lightweight, lower strength materials with strong fibers or particles. This concept is the central idea in composite materials [2-4]. Materials of high strength-to-weight ratios are especially attractive. Normally, the fibers have high strength and elastic modulus, while the matrix material is ductile and weak. Also, the matrix must be non-reactive with the fibers. Metals and polymers have been used as matrix materials, and glass, boron, graphite, and metal wires, such as tungsten, have been used as reinforcement[5]. The fibers may be long and continuous, or relatively short and discontinuous. The most common reinforced composite materials are the glass-fiber reinforced products[6].

In fiber reinforced composites, the matrix serves to transmit the applied load to the fibers, protect fibers from damage, and to separate the individual fibers. The high modulus fibers carry essentially all of the load [7] through shear stresses developed along the fiber-matrix interface[8].

Silicon carbide whiskers produced from rice hulls were first reported in 1975 [9]. The promise of the material at that time was recognized to be the potential low cost since the whiskers were the derivative or a waste product. During the past few years, SiC_w have been introduced into aluminum matrices to form composites.

Data on the mechanical properties of the SiC whiskers in various Al alloys are given in a paper by Divecha, Fishman and Karmarker [10], and in the minutes of the Fourth Annual Discontinuous Reinforced Aluminum-Material Working Group Meeting [11]. The most complete study for a particular alloy has been one conducted by ARCO Metals for the Naval Surface Weapons Center (Dahlgren, Va.) [12]. This study concluded that Al 6061 alloy containing 20 v/o SiC whiskers was a very promising and reproducible system. The material showed little variation in whisker uniformity in different extrusions, and reproducible tensile properties were obtained with little scatter. The mean values obtained for 60 tests were:

Elastic Modulus = 118.6 GPa (17.2×10^6 psi)

Yield Strength = 465 MPa (66.2×10^3 psi)

Ultimate Strength = 600 MPa (87.0×10^3 psi)

Elongation = 1.98 %

These values represent increases of 72%, 114%, and 149% for the modulus, yield, and ultimate strengths, respectively, and a decrease of 89% in the elongation to failure over a typical 6061/T6 alloy. In spite of the very favorable tensile properties, it is the decrease in elongation (or strain to failure) that is one of the primary problems in this composite system.

Marcus [13] in a study of the whisker-matrix interfacial region using Auger techniques, has determined that

interfacial bonding is basically good in the system. He further states that the oxide Al_2O_3 and intermetallic beta-Al_4SiC_4 were present in the interfacial region. Marcus concluded that the interface does not play a major role in the fracture of the material, because fracture occurs primarily in the matrix. More interfacial composition studies have been made by Nutt and Carpenter [14], where MgO has been detected at the interface.

That the whiskers themselves contribute to composite strength is not shared by all investigators. Sanders states that fiber volume fraction, distribution, and length-to-diameter (L/D) ratio, along with other parameters, control the density and distribution of matrix dislocations. An unfavorable distribution of fibers and dislocations can result in premature failure due to strain localization [15]. Sanders conducted a study of aluminum SiC whisker and particulate composites [15]. He concluded that since strengthening mechanisms were similar for all reinforcement morphologies examined, the aspect ratio of the whiskers was apparently insufficient to alter the strengthening mechanism. However, quantitative L/D values were not reported.

Arsenault and Fisher [16] suggest that primary composite strengthening is attributable to the high density of dislocations which are generated when the material is cooled during fabrication. Formation of these dislocations is due to the thermal expansion mismatch between the matrix and

whiskers. They further state that the discontinuous whiskers are not of sufficient dimensions to support a load, and that virtually none of the whiskers have the length-to-diameter ratio of 66 necessary for strengthening. This theory, however, does not account for the observed anisotropic strength properties of the composites [17], where transverse tensile strength is approximately 75-80% of the longitudinal value.

Using the relationship developed by Kelly and Tyson [18], that is:

$$L_c/D = S_f/2\tau_m$$

where: L_c = critical fiber length to achieve maximum stress in the fiber

D = fiber diameter

S_f = maximum fiber tensile strength

τ_m = shear yield stress of the matrix (or interfacial shear strength, whichever is lower)

Wawner [19] has calculated the critical L/D ratios for assumed whisker strengths. The matrix, 6061-T6 Al, has a shear strength of 30 ksi (207 Mpa). An indication of Silag whisker strength can be obtained by extrapolating the curve of composite strength vs. volume fraction of whisker to 100% volume fraction (figure 1). Although not completely valid, because of whisker misalignment, the value obtained should represent a lower bound. As shown in the figure, the

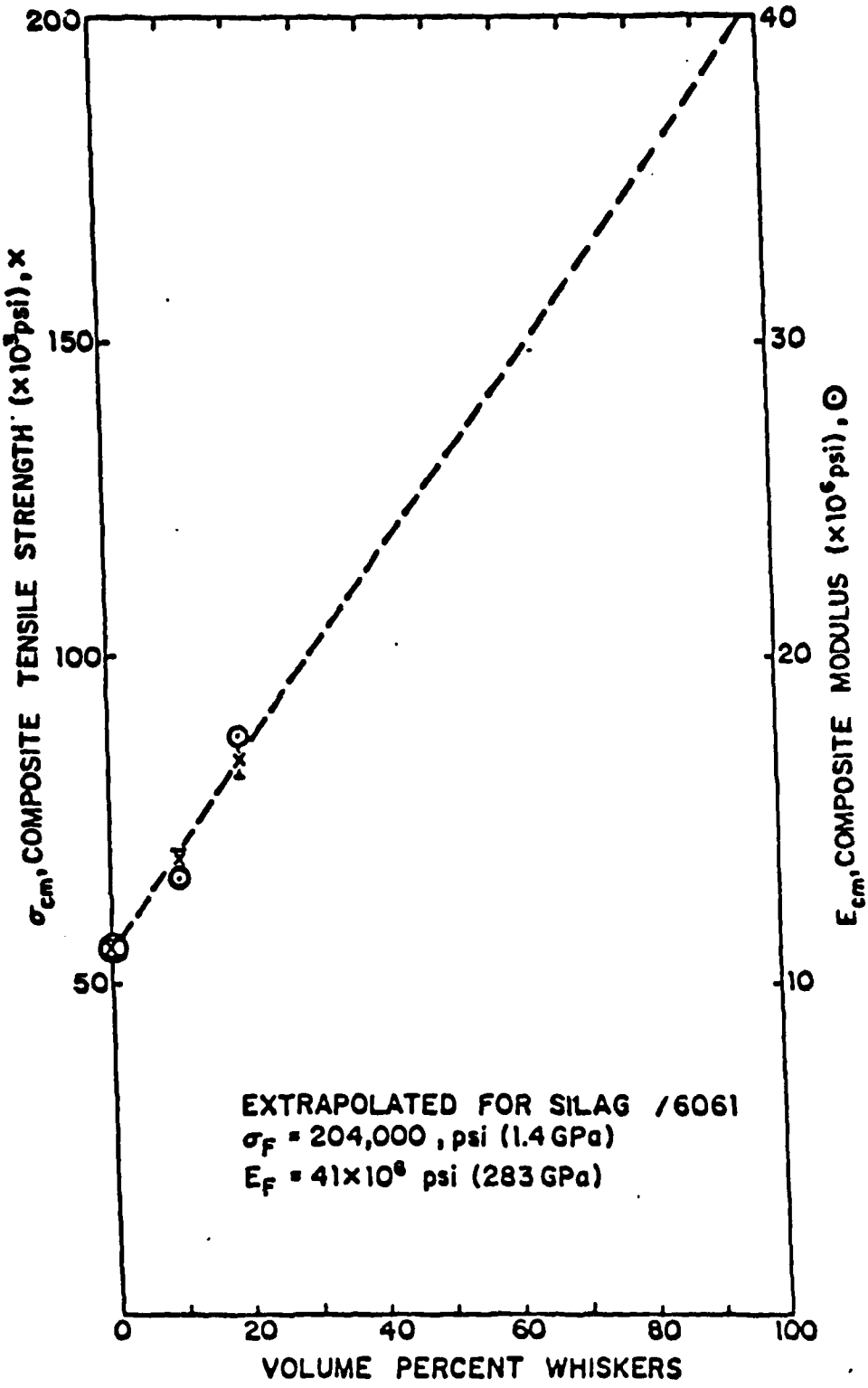


Figure 1. Tensile Strength vs. Volume % Whiskers [19].

extrapolated value is 204 Ksi (1.4 GPa), a reasonable number for imperfect whiskers with surface defects. From this value then, a critical length-to-diameter ratio (L_c/D) of 3.4 would be obtained for the Silag - 6061/T6 system. If whisker strength is assumed to be 1000 Ksi (6.9 GPa), a value that is not unusual for small diameter whiskers, then a L_c/D ratio of 16.7 would be obtained for the system.

Work by Nardone and Prewo [17] has provided further support for the theory of load transfer to whiskers. In their study, a modified formula for critical fiber length was developed, and tested with 6061-SiC_w composites. The modification takes into account transfer of load at the ends of short fibers. Other theories neglect fiber end effects. Nardone and Prewo state that it is possible to obtain strengthening from whiskers with aspect ratios of $L/D = 3$. Their results are in good agreement with the above calculation of $L_c/D=3.4$.

At the University of Virginia, Wawner and others [20], have determined several factors that influence fracture:

1. Incomplete powder compaction during composite preparation results in weaker interfacial bonding, and porosity.
2. Matrix alloy chemistries can effect precipitation of large, brittle particles, which can acras crack propagation paths.
3. Nonuniform whisker distribution results in

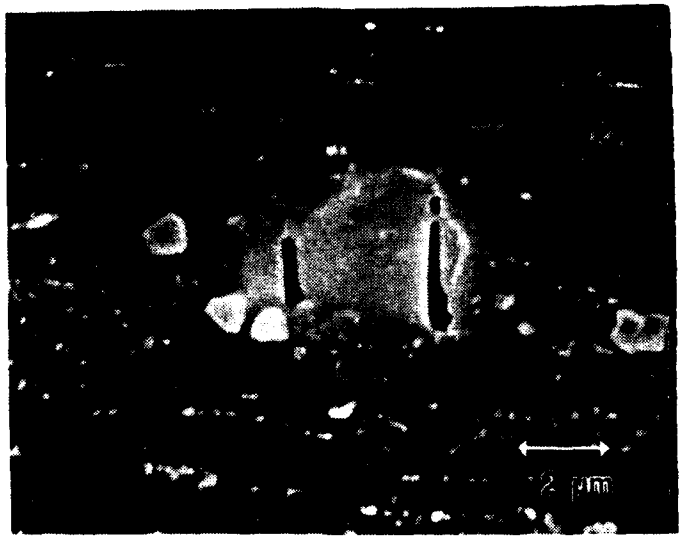
fiber- fiber contact. These contact areas are sites for precipitation of brittle particles.

4. Impurities present in raw materials (Fe, Mn), also contribute to precipitate formation.

5. Variation in whisker diameter can inhibit uniform distribution of reinforcement throughout the matrix.

Figures 2 and 3 depict the brittle nature of large precipitates in 2124 Al specimens which were prepared by identical powder metallurgy techniques as the composite, and tested in tension [21]. Although the surrounding matrix is relatively undeformed, large cracks are clearly seen to propagate through the particles. Energy dispersive spectroscopy (EDS) has revealed Mn and Fe content in these precipitates.

Fracture toughness measurements by Harrigan [22], made on SiC particulate reinforced 6061 Al, have shown K_{1c} to be a function of SiC content and heat treatment conditions (Figure 4). In this figure, not only are values for composites given, but also data for some standard Al alloys are presented for comparison. The alloy materials all have higher toughness values than do the composites. This fact can be understood by considering toughness as the ability of a material to absorb energy in the plastic range[23]. In terms of a stress-strain diagram then, the area under the curve is an indication of the amount of work done on the material, or the amount of



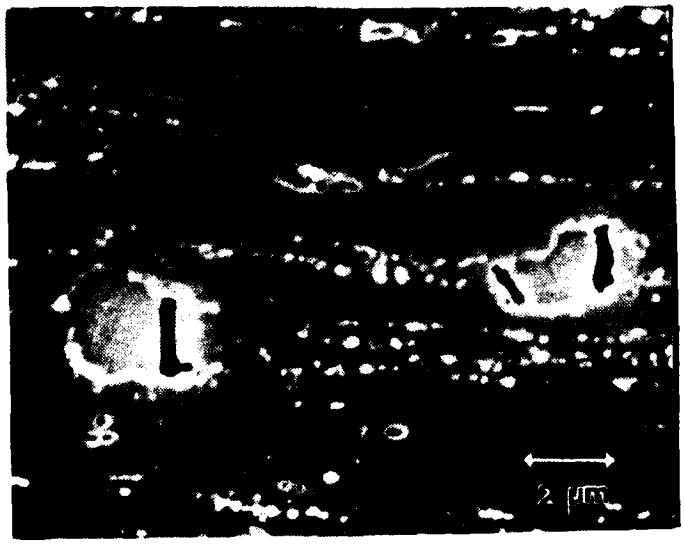


Figure 9. Set of A - W Containing Particles in 2124 [illegible]

Figure 3. TEM of Al-Cu Containing Particles in 2124 Tensile Specimen [21].

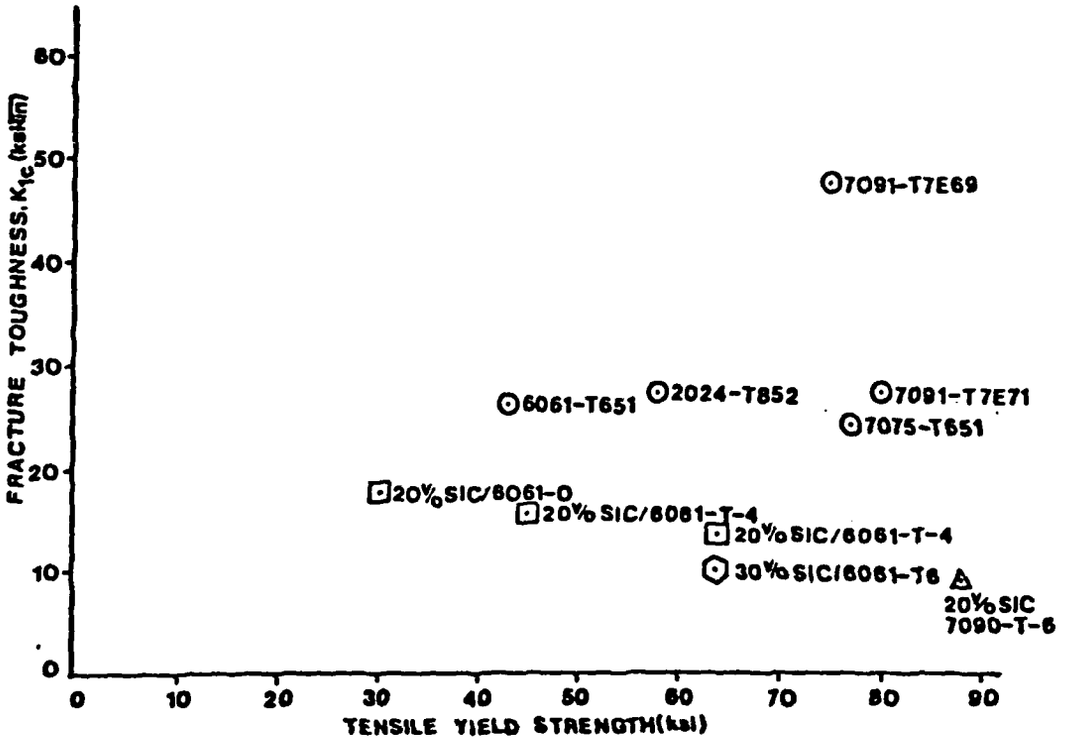


Figure 4. Fracture Toughness vs. Tensile Yield Strength [22].

energy absorbed (see Figure 5). The incorporation of SiC whiskers into the matrix increases the tensile strength, but greatly decreases the elongation. Thus compared to the alloy alone, the energy absorbed in composites is much less.

In Figure 4, it can also be noted that composite toughness decreases with increasing yield strength. Higher strength is achieved by transfer of stress from the matrix to the fibers via a strong fiber-matrix interface [24]. A stronger interface implies higher strength. Concomitant with a stronger interface, however, the ductility of the matrix is further restricted and elongation decreases. Hence, fracture toughness declines. The competing effects of higher strength at the expense of ductility is the major problem inhibiting widespread use of composite materials.

Bettadapur and Wawner [25] conducted a study of tensile strength as a function of temperature for 6061 Al-SiC composites. Materials were heated and hot pressed at different elevated temperatures, then mechanically tested at room temperature. As shown by the results in Figure 6, ultimate tensile strength varies after exposure to different temperatures. It is interesting to note that, generally, as treatment temperature increases, strength values decrease in magnitude, then show an increase in strength again. (The top curve is an exception.) Possible influences are temperature, hot pressing effects, or some combination of the two. There was, however, no definite explanation given for this

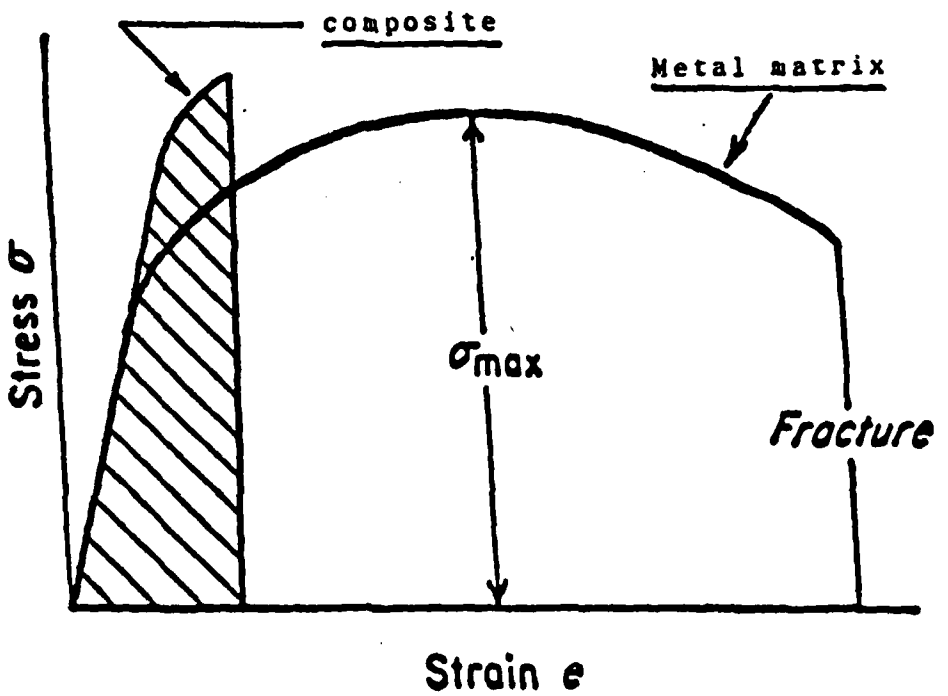


Figure 5. Comparison of Composite and Metal Matrix Stress-Strain Diagrams.

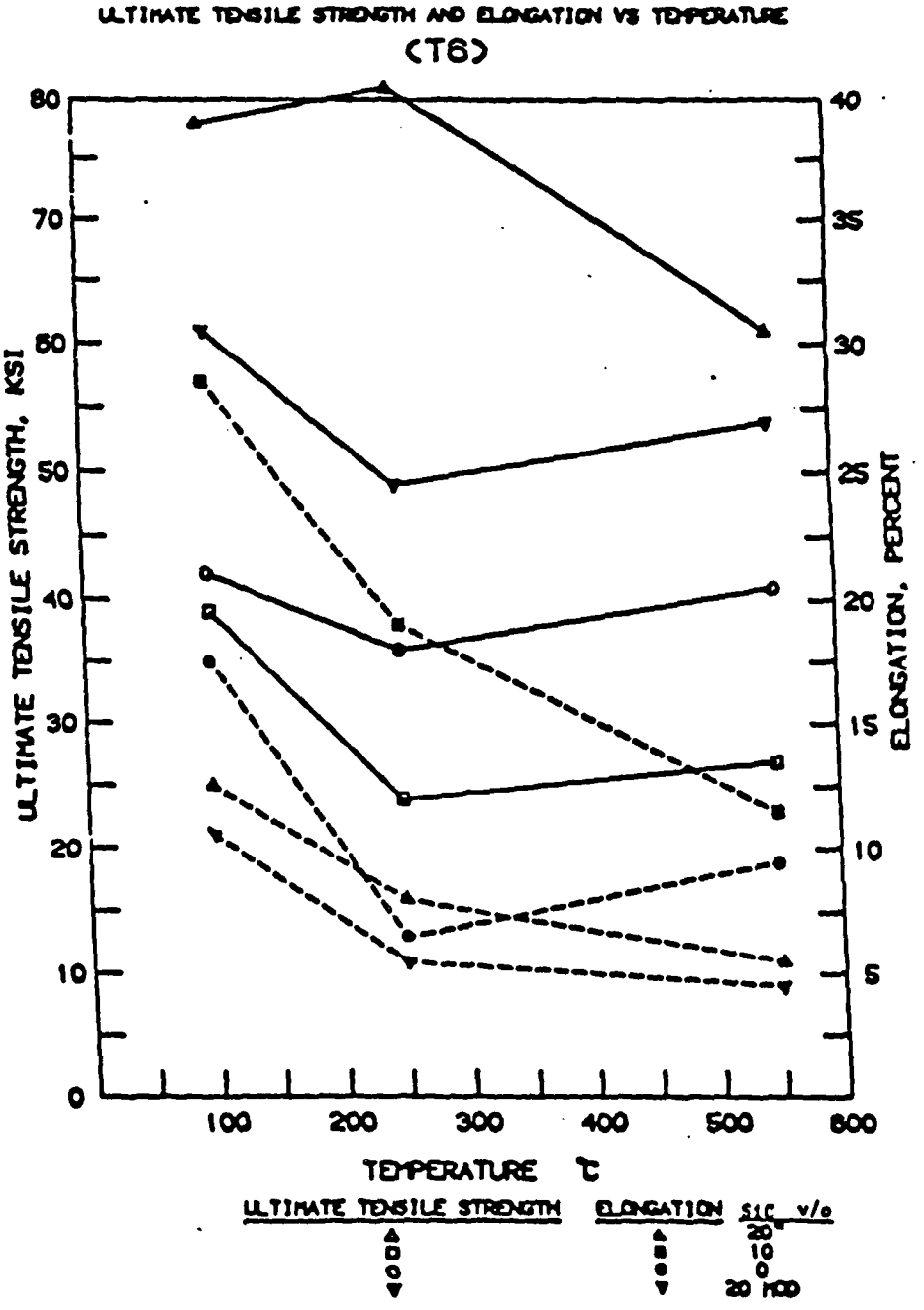


Figure 6. Ultimate Tensile Strength and Elongation vs. Temperature [25].

observation.

Fracture surfaces in Al-SiC_w show fine matrix dimples [26] which are indications of ductility, although on the macroscopic level, the materials behave in a brittle manner. Whiskers without aluminum adhering to them, have been found on fracture surfaces. This phenomenon indicates that debonding at the interface is a common feature in fracture [27]. Webster [28] also suggests that interfacial bonding can be an important factor in composite strength.

Fracture studies by Wawner [21] clearly show the importance of the interface. Tensile fracture specimens of 2124-SiC_w composites were examined with TEM (Figures 7 and 8). Cavities are found to form at whisker tips and along the interface. The same phenomenon of cavity formation was also reported by Nutt and Duva [29]. Any weak bonding between matrix and whisker can cause premature crack initiation in these areas. Correlation of mechanical properties with microstructure in investigations such as these can lead to strengthening theories. However, many fracture studies have not provided detailed microstructural characterization of test specimens, and so application of general theories is difficult [30].

Deformation studies of composites have yielded conflicting results. Brewer and Sarkar [31] found that hot rolled 2124/SiC_w sheet had higher strength than extrusions or rolled plate. They attribute their findings to better

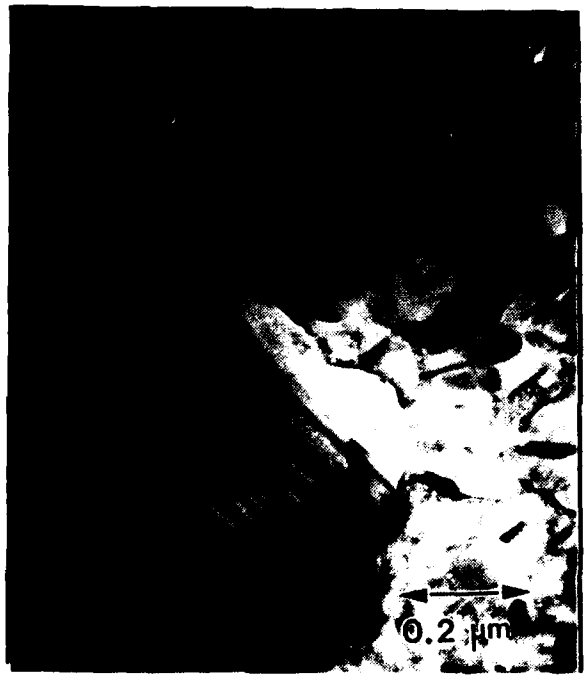


Figure 7. TEM of deviation at whisker tip in SiC_w [10].

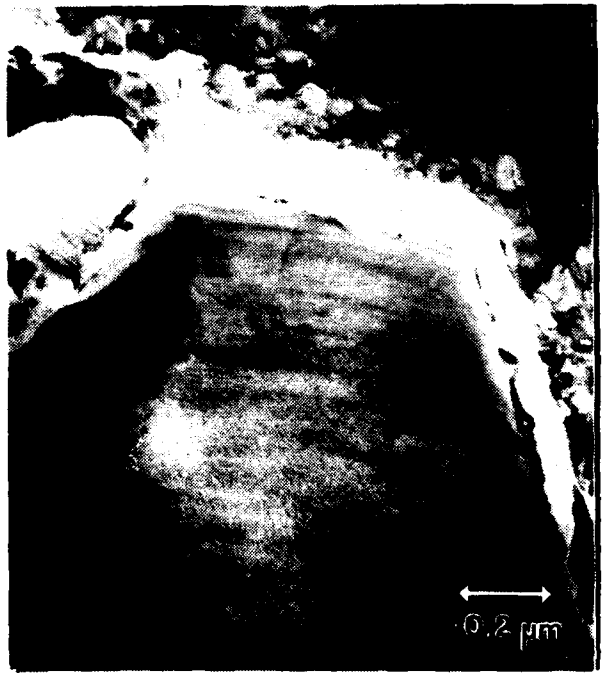


Figure 8. TEM Showing Cross Section of Whisker with Interfacial Failure [21].

homogenization of alloying elements, and better particle distribution, induced by the rolling process. In the same report, however, they state that rolling caused a 10% reduction in modulus in the extrusion direction of the material. Harrigan and others [32] found that strain to failure is a function of roll reduction. They also showed that hot rolling of 6061 composites to greater than 80% reduction in thickness, improved both tensile strength and strain to failure values (see Figure 9). In contrast, Nieh and Karlak [33] demonstrated in 6061 and 2024 composites, that properties of hot rolled materials were comparable to extrusions, although the hot rolling resulted in more uniform microstructure. In a thermomechanical study of 7475 alloy/SiC composites, Ghosh et.al. [34] also conclude that there is not a significant difference in the effects of secondary hot rolling or forging on composite properties.

Heat treatments for unreinforced Al alloys have been characterized in depth [35,36]. Heat treating encompasses any heating or cooling operations performed on an alloy in order to change mechanical properties, structure, or stress state in the alloy. With aluminum alloys, an increase in strength and hardness in precipitation hardenable materials is the usual objective of such treatments. Normally, a three part process is employed to achieve higher strength:

1. Solution heat treat the alloy at a temperature high enough to dissolve the soluble phases.

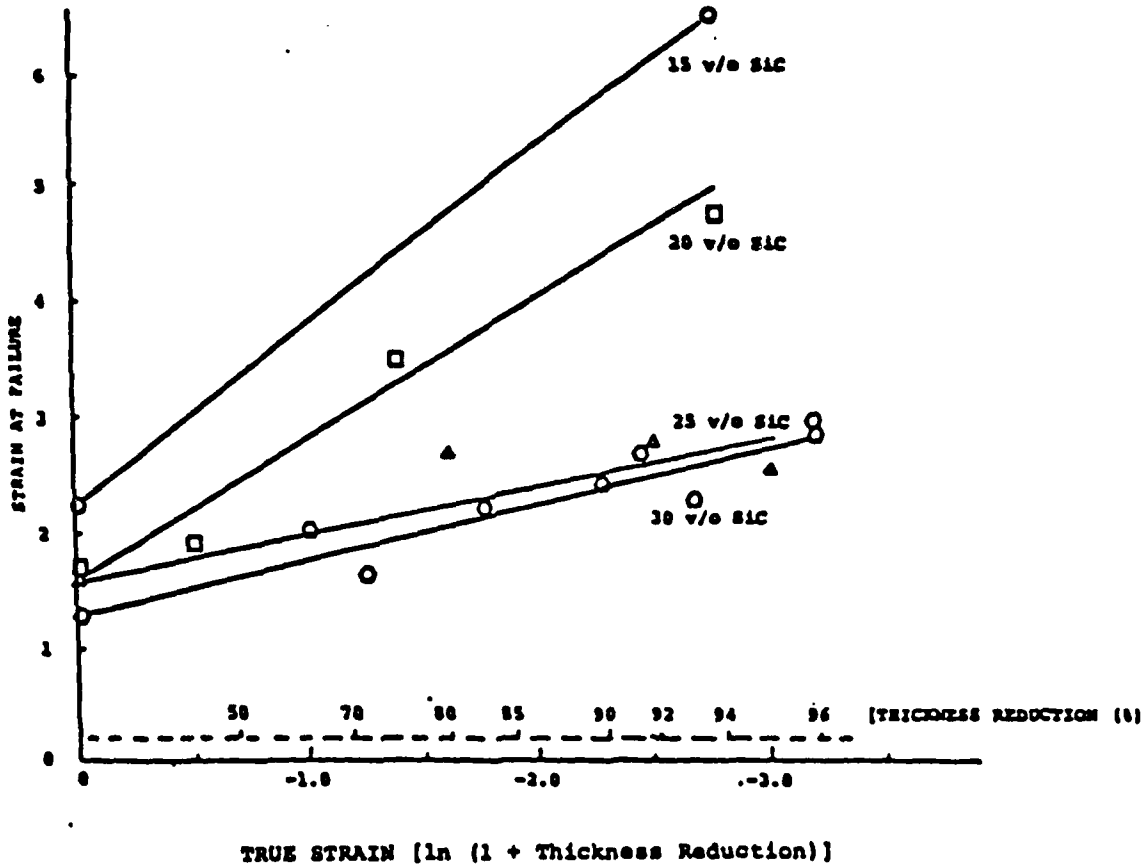


Figure 9. Graph Showing Strain at Failure Dependency on Roll Reduction [32].

2. Quench (usually rapidly) from the solution temperature, in order to develop a state of supersaturation.
3. Age the material to precipitate the strengthening phase, either at room temperature (natural aging), or at elevated temperature (artificial aging).

Times and temperatures used in these processes vary with each particular alloy composition, and the properties desired.

When composite materials using Al alloys were first being developed, it was natural to heat treat according to methods developed for the matrix aluminum. And so, most thermal treatments for aluminum composite materials are based on studies of the pure matrix alone. However, using differential scanning calorimetry, Papazian [37] has shown that the presence of SiC in a 6061 Al matrix can modify precipitation behavior, as seen in Figure 10 . Therefore, composite heat treatments based on matrix information alone, may not be yielding optimum results.

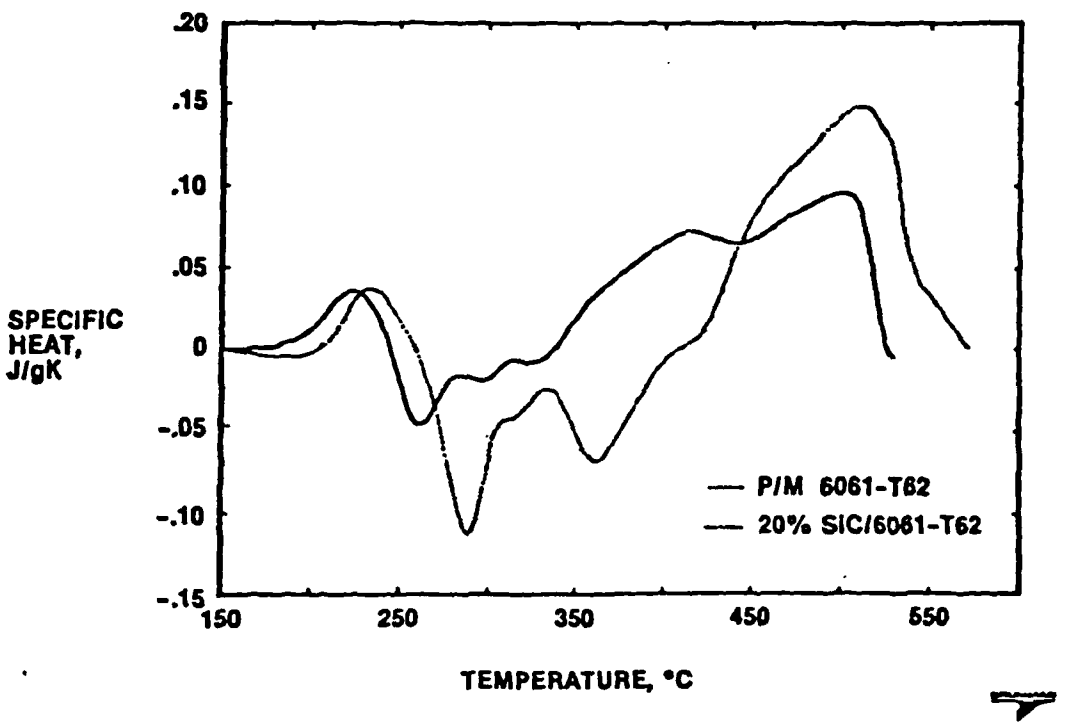


Figure 10. Differential Scanning Calorimetry Curves for 6061 Matrix and Composite [37].

EXPERIMENTAL PROCEDURES

Material Acquisition:

All materials investigated were fabricated by ARCO Chemical Co./Silag Operation in Greer, South Carolina, according to the following general procedure (see Figure 11), the exact details of which remain proprietary:

1. Loading of the blended aluminum alloy - silicon carbide whisker material into a die.
2. Loading the die into a vaccuum hot press.
3. Heating the loaded die while alternately evacuating and backfilling the chamber with an inert gas.
4. Heating the loaded die to a temperature above the alloy solidus under vaccuum.
5. Applying pressure at a controlled rate until fully dense.
6. Cooling the die and removing the billet from the die.
7. Extruding the billet at an elevated temperature into 2.5 cm thick plate.
8. Materials received in rolled sheet were hot rolled at temperatures just below the solidus. Successive passes of 10% reduction were used to obtain the desired thickness.

Microstructure Characterization:

Three general subdivisions of the composite - matrix, whiskers, and fiber-matrix interface, were examined with scanning and transmission electron microscopy (i.e. SEM and

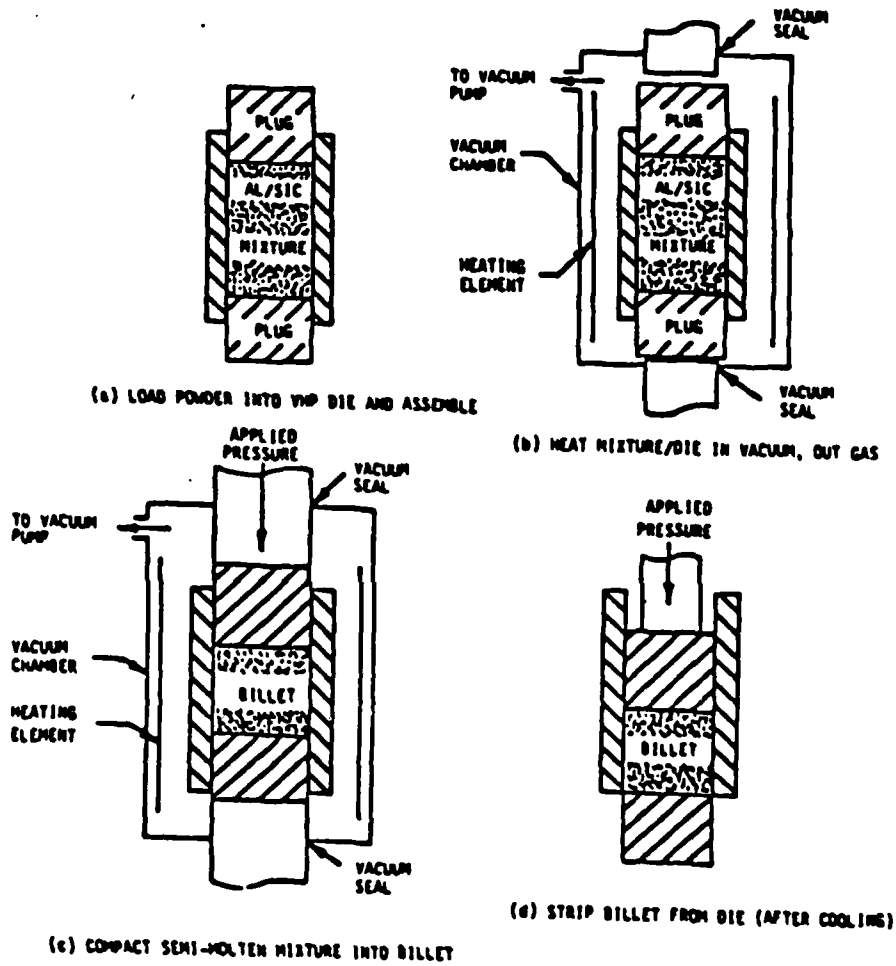


Figure 11. Schematic of Vacuum Hot Pressing Operation [40].

TEM), to note any changes occuring after thermomechanical treatments.

Cross sections of as-received material were ground with 400 and 600 grit emery cloth, then polished with 6,3, and finally 1 micron diamond paste using a napless cloth. Final polishing was done with 0.05 micron MgO solution, which also served as a slight etchant, enhancing whisker, precipitate, and porosity observation.

Polished surfaces were first sputter coated with an approximately 100 angstrom thick layer of gold to improve electron beam conduction at the whiskers, then examined with scanning electron microscopy using a JEOL model JSM-35 microscope to characterize the following features:

1. Distribution of matrix components - precipitates, inclusions, porosity , and cracks.(The sizes and locations were noted, and composition was studied using enegy dispersive x-ray spectroscopy.),
2. Whisker size, distribution, and alignment throughout the matrix.(Also, any irregularities, such as damaged whiskers were investigated.).
3. The interfacial region, i.e. the occurrence of precipitates or pores (non-wetting effects) along the interface, or the absence of these phenomena were studied.

Further investigation of whisker length-to-diameter (aspect) ratio, and also the amount of porosity, was done

using a Lemont Scientific Corporation image analyzer. SEM micrographs were used as input to the machine. The particles of interest (whiskers or pores) were then analyzed automatically using a chord length algorithm to determine size (length and diameter for whiskers) or volume fraction (for pores).

Gold coated fracture surfaces were examined in the SEM. Fibers, matrix, and interface again were analyzed for fracture origination and propagation. Comparison of microstructure, before and after tensile testing was made.

A Philips EM 400T microscope operated with an accelerating voltage of 120 kilovolts was used for transmission electron microscopy studies. As-received materials were examined to characterize matrix, fiber, precipitate, and dislocation morphology. Differences in microstructure before and after treatments were recorded.

Tensile Testing:

An Instron model TTCML tensile testing machine was used to perform tensile tests. A 500 kilogram load cell was used with a crosshead speed of 0.05 cm/min. Specimen dimensions are shown in Figure 12. Limitations imposed by the dimensions of extruded composites, and also by rolling parameters, necessitated deviation from usual ASTM standards. Samples were first cut to the desired length as 3 mm square bars. The gauge length was then reduced to a 3 mm diameter cross section using a metal lathe, and sanded to a 600 grit finish

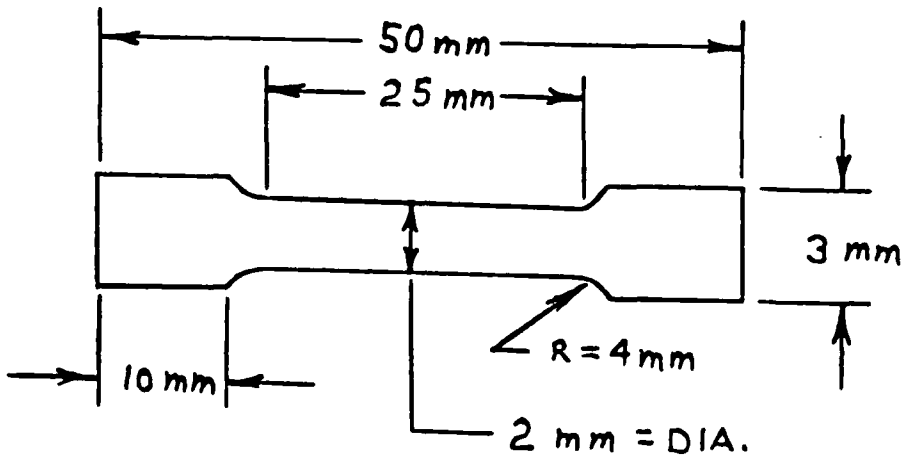


Figure 12. Tensile Specimen Specifications.

with emery cloth.

Rolling

A Fenn 5 inch diameter rolling mill was used to roll materials at a rate of 16 feet per minute. Samples were hot-rolled at 493^{O} C and air-cooled. No quenchant was used. Reductions in thickness of 10% were made with each pass through the rollers. The samples were reheated at 493^{O}C for 15 minutes between each pass. Starting specimen dimensions were approximately 1 cm thick x 1.2 cm wide x 5 cm long. The rolling direction was chosen to be perpendicular to the extrusion direction. This avoids damaging the whiskers, which tend to align lengthwise along the extrusion direction.

Differential Scanning Calorimetry:

Transitions, such as melting, precipitation, and dissolution, were investigated with a Perkin-Elmer Model DSC-7 differential scanning calorimeter (DSC). Three mm diameter by 1 mm thick specimens were heated in a nitrogen atmosphere at a rate of 20^{O} C/minute, starting at 50^{O} C and going to final temperatures of between 500 and 650^{O} C, depending on the matrix material being investigated. Reaction temperatures were determined from plots of milliwatts (i.e. energy units) vs. temperature.

Heat Treating:

Reaction temperatures identified with DSC (220^{O},270^{O},450^{O}, and 493^{O}C) were used as heat treatment

temperatures for both 1100 and 2124 matrix - 20 v/o SiC_w composites. Samples were heated for 1 hour at the temperature of interest, cold water quenched, and then aged for 10 hours at 160° C.

Both extruded and rolled materials were prepared in the identical manner to see if heat treatment had any significant effects in the deformed vs. the undeformed material.

TEM Sample Preparation:

Three mm diameter discs were cut from bulk material and ground to a thickness of 0.005 in. with 600 grit emery cloth. These discs were further polished using 6 micron diameter diamond paste solution on a VCR Group Dimpling machine, to a thickness of 0.002 in. Final polishing to perforation was done with argon ion-milling using a Gatan Model 600 dual ion-mill, at settings of 4 kilovolts accelerating voltage, 1.0 microampere current, and a final ion stream angle of 5° inclined to the specimen surface.

Results and Discussion

Description of Research:

Past microstructural studies have indicated that constituent particles in the 3-5 micron size range dominate failure in the present composite systems of interest (i.e. SiC/2124 and SiC/6061). These particles, identified through x-ray analysis as: Al_2CuMg, $Al_{20}Mn_3Cu_2$, and $FeCuMnAl_6$ [38], influence fracture toughness since they generally are brittle and form in critical locations such as whisker-matrix interfaces or along grain boundaries. These particles are also found to form in areas where whiskers are in contact, as shown in Figure 13. This figure is a TEM micrograph of a precipitate formed at the contact area of two whiskers in a 2124 matrix composite. Figures 14 and 15 show SEM micrographs of typical particles on the fracture surfaces of this material. Included is an energy dispersive x-ray spectra indicating that Cu, Fe, and Mn are found in these particles. The particles exhibit cleaved surfaces compared to the ductile appearance of the surrounding matrix. Also seen in Figure 15, is a crack passing through the particle. Pore formation can also be seen.

Minimizing the effects of these detrimental particles is a first step toward improved fracture toughness. Cleaner whisker starting material and improved extrusion methods developed at ARCO Silag, have greatly improved whisker distribution throughout the matrix. In Figure 16, note the

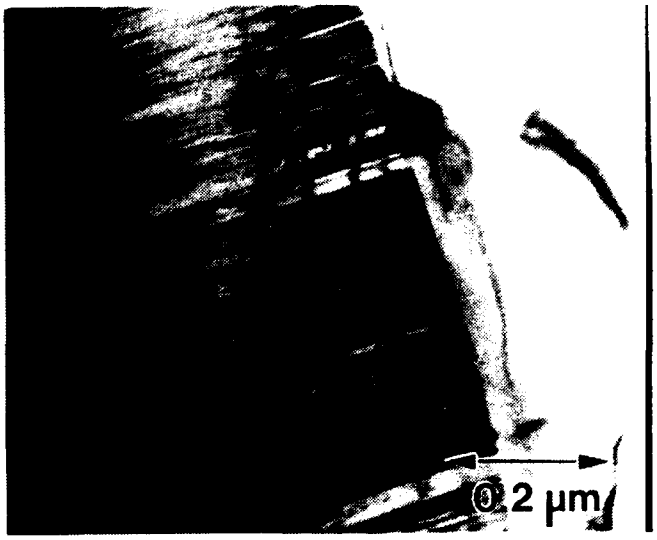
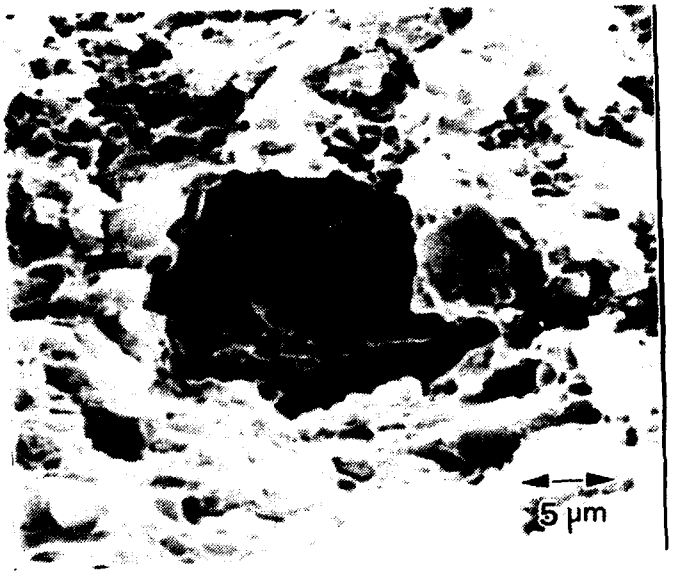


Figure 13. Precipitate Formation Along Interface and at Whisker-Whisker Contact Point



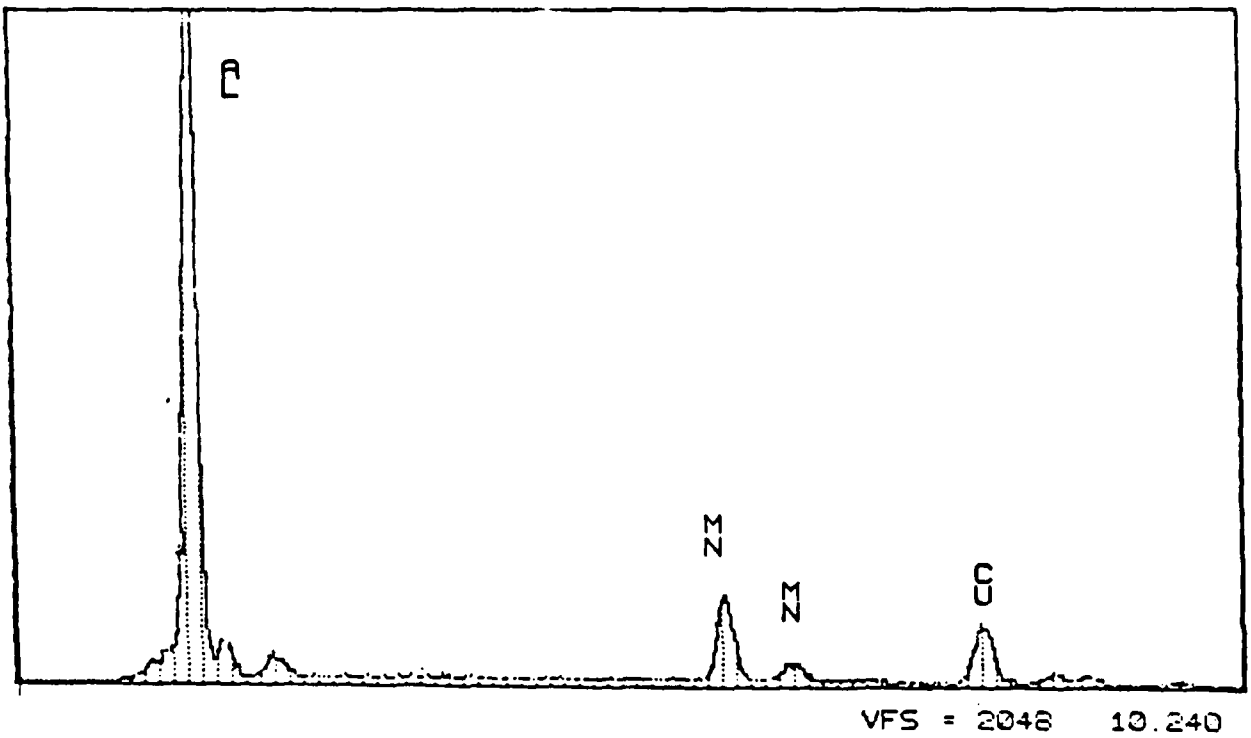


Figure 14. Brittle Intermetallic in 2124-SiC_w Composite and Corresponding X-Ray Spectrum

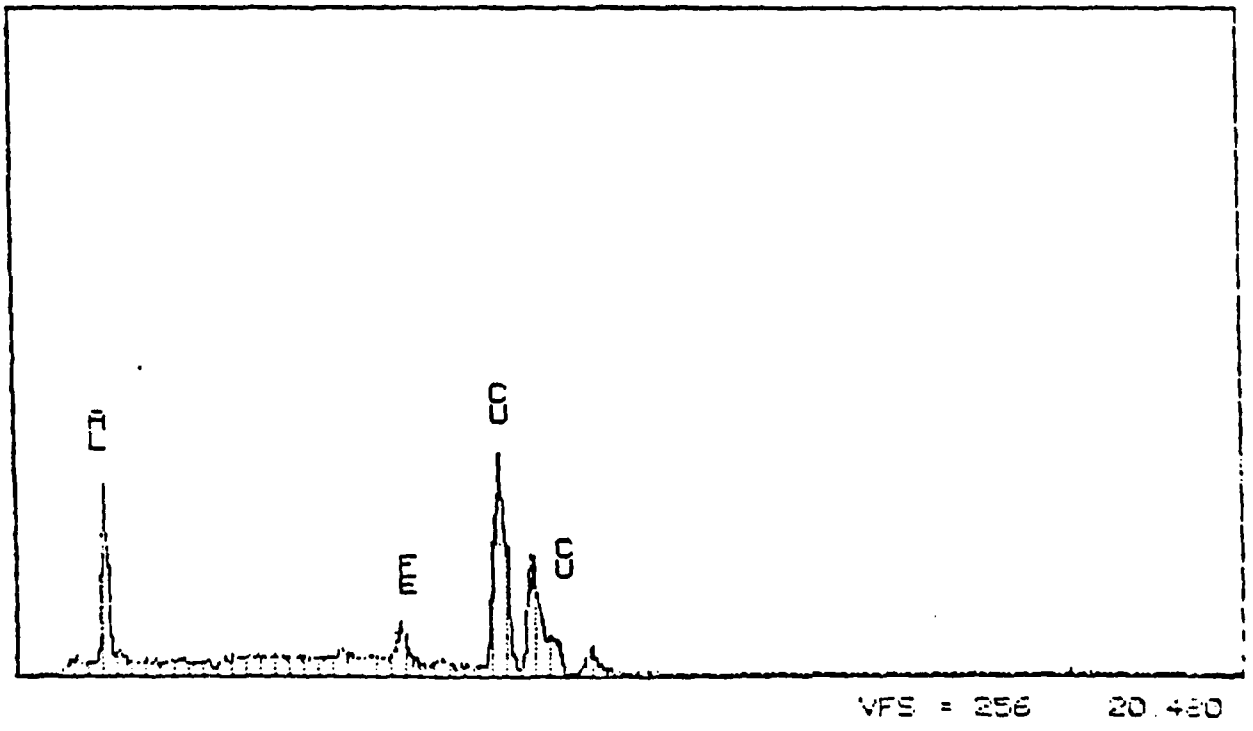
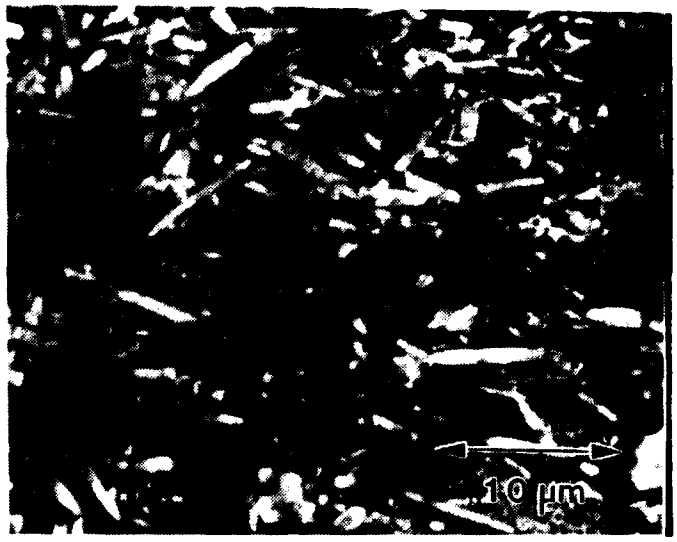


Figure 15. Brittle Intermetallic in 2124-SiC_w Composite and Corresponding X-Ray Spectrum



A

B

Figure 16. Whisker Alignment in (a) "Older 2124", and (b) 2124 With Improved Extrusion

distribution and orientation of whiskers in "older" 2124 in Figure 16(a), compared to the more recently processed material in Figure 16(b). With better whisker distribution, the probability of precipitate formation at whisker-whisker contact areas is greatly reduced. In addition to minimizing precipitate formation sites, matrix alloy chemistry can be altered to reduce the amount of matrix alloying elements present (i.e. Fe,Mn,Mg,Cu), which are the major components of these precipitates.

New alloys of differing Cu and Mg content (and also a Li-containing alloy) were used to form composites with SiC_w. The compositions and fiber volume fractions are listed in Table I. These three composites, along with 1100-20 v/o SiC_w and 2124-20 v/o SiC_w are the materials used in this investigation. Less than 50 grams of each of the new alloy composites was provided by ARCO, therefore only very limited thermomechanical testing of these new materials was performed. Most experiments were done with 1100 and 2124 matrix composites.

General Microstructure of As-received Materials:

The basis of metal matrix composite strengthening is the addition of a high modulus reinforcement to a lower modulus matrix material. There is a critical aspect ratio for the fibers which must be exceeded to fully utilize the high strength properties of the reinforcement [24]. The aspect

Table I.

Compositions of Composites Being Studied

Matrix	Reinforcement
Al - 1.44 w/o Mg	20 v/o SiC_w (F8)*
Al - 0.85 w/o Mg, 0.91 Cu, 1.66 Li	15.9 v/o SiC_w (F8)
Al - 1.37 w/o Mg, 2.95 Cu	19.3 v/o SiC_w (F8)
1100 Al	20 v/o SiC_w
2124 Al	20 v/o SiC_w

* F8 is the quality grade designation for silicon carbide whiskers produced by ARCO Chemical Co.

ratios of all materials listed in Table I were measured, in order to determine if, not only damage incurred during the handling and processing of SiC_w was affecting final aspect ratio, but also if other factors such as formation of precipitates, volume fraction of the fibers themselves, and other component interactions, also have an influence.

Processing conditions of all composites were held constant while matrix composition and whisker volume fraction were varied. Figure 17 is a typical aspect ratio histogram obtained for all 5 systems tested. The aspect ratios are basically the same in all composites investigated. The most probable ratio is 3 to 1 in each instance. Slight volume fraction and matrix variations have no major effect on aspect ratio.

As seen in Figure 16, the new composites exhibit much improved whisker alignment and distribution. Since surfaces for SEM examination of whisker distribution were polished and etched, it was necessary to verify that polishing was not influencing orientation. TEM samples were examined, and found to exhibit the same fine distribution of whiskers, as shown in Figure 18.

The number of large (3-5 micron size) particles found in 2124 and 6061 composites, has been drastically reduced by lowering the Cu and Mg content of the new matrix alloys, and also by using 'cleaner' raw materials (i.e. less impurities such as Fe and Mn in the starting material powder). Again, in

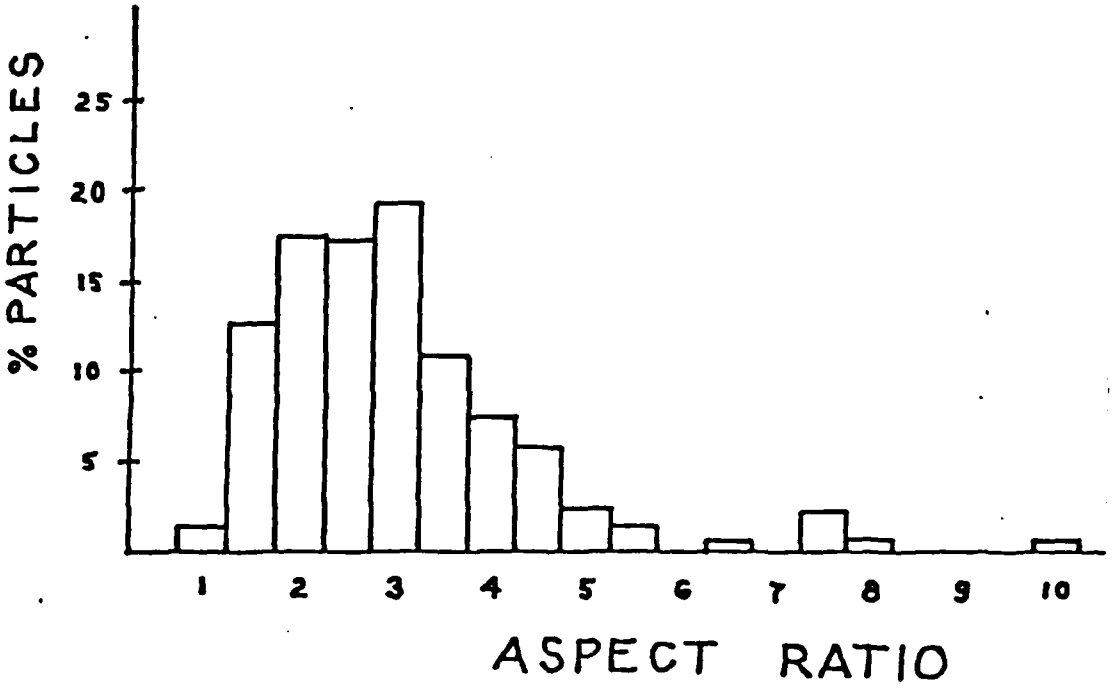


Figure 17. Typical Aspect Ratio Histogram Obtained For All Composites Tested

Figure 16. TEM of Blue Whisker Distribution and Alignment After Improved Fabrication Techniques

Figure 18, the relatively clean matrix is evident. Very few precipitates of sizes larger than whisker diameters (0.5 micron) were found. Attempts to x-ray map areas of Cu and Mg using EDS produced no areas of significant concentration in the new matrix alloys. There are, of course, still some large intermetallics formed, as seen in Figure 19, but they are not very common.

Figure 19 also shows large pores. There is still a considerable amount (1-2% as measured from SEM micrographs on the image analyzer) of porosity in the compacted composites. Figure 19(b) shows a higher magnification micrograph of the pores. To further reduce porosity, more improvement in the powder metallurgy techniques used for composite processing is necessary .

Matrix-whisker interfacial areas are generally very clean in the Al-Cu-Mg and Al-Li-Cu-Mg alloy composites [see Figure 20(a)]. In the Al-Mg binary alloy, however, precipitates are seen to form along the interface [Figure 20 (b)]. The large number of brittle precipitates, as previously seen in 2124 and 6061 composites, has been greatly reduced in these new alloys, thus resulting in cleaner interfaces.

Dislocations have been observed at the SiC fiber ends and running through grains adjacent to whiskers (Figure 21). Dislocation density measurements were not made, but intense tangles, or forests, are generally observed in the undeformed materials.

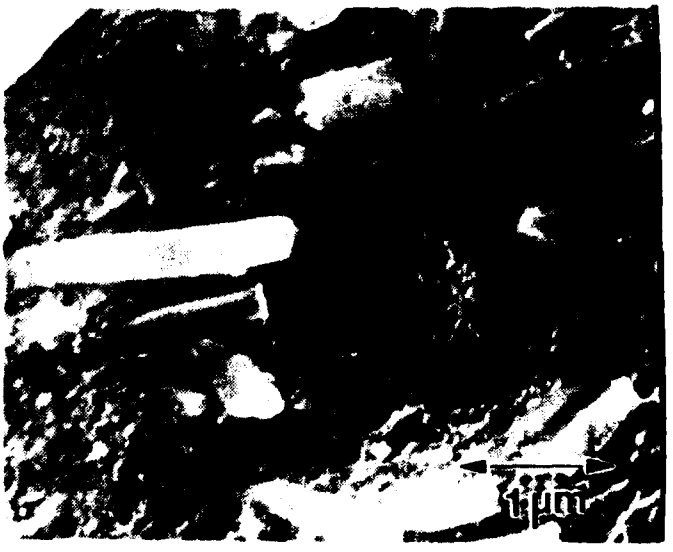


Figure 11. Large Intermetallic Particle and Porosity in Alloyed Alloy Composites

Figure 20. [illegible]

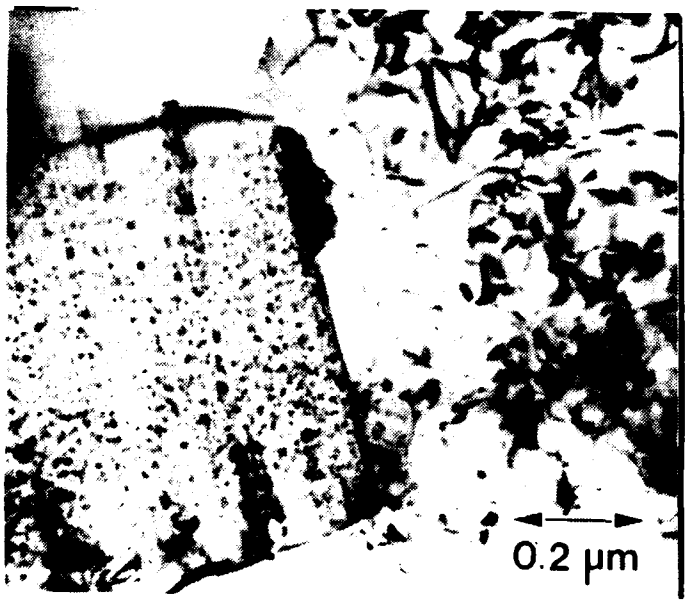


Figure 21. Dislocations at the end of a whisker.

Importance of Whiskers and Interface:

As discussed previously, there is presently a question as to whether or not the aspect ratio (L/D = 3) of the SiC_w used in these composites meets the critical aspect ratio criterion for fully utilizing the fiber's strength. Another important issue is whether or not the whiskers, even if they possess the required dimensions, actually do carry a load. It is also possible that the increase in matrix dislocation density due to the presence of the fibers, is the major strengthening mechanism.

Sections of fractured tensile specimens of 2124 and 1100 composites were examined using TEM. Figures 22(a) and (b) are electron micrographs depicting SiC whiskers which have fractured under the applied tensile load. The stress axis was parallel to the whiskers' major axes of orientation. Areas devoid of matrix material can be seen between the corresponding sections of the fractured whiskers. If the whiskers had broken during fabrication, the region between these sections should have been infiltrated with matrix material. Also, examination of underformed composites revealed no similar whisker damage or cavitation between whiskers, although on a much larger scale, some porosity is present.

It is interesting to contrast the cavitation in the latter micrographs with that which was shown in figures 7 and 8. Clearly, a weakly bonded matrix failed at the interface, while strong and well bonded to good stress transfer to the

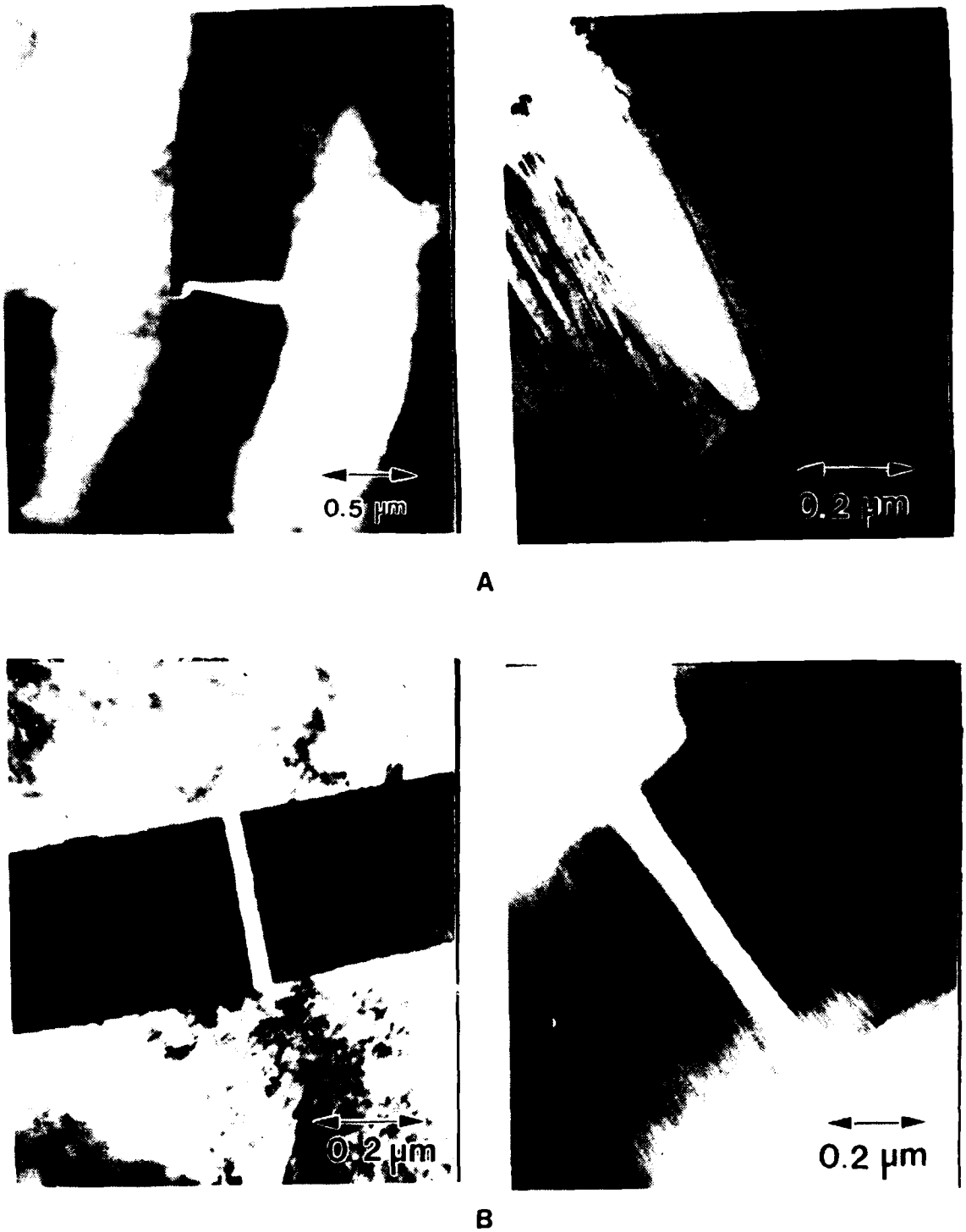


Figure 22. Fractured Whiskers in (a) 7100, and (b) 7104 Fractured Composites

reinforcement. Fracture then occurs within the fiber itself, not at the interface. Therefore the material is stronger.

The most probable explanation for the observed whisker fracture is that the tensile load has been transferred from the matrix to the whiskers, which can then act as the major load carrying mechanism. As shown in Figure 22, fracture usually occurs in the central region of the whisker length, where maximum stress within the fiber occurs. Any defects along the whisker length can, of course, shift the crack initiation site away from the center of the fiber, but this was not generally observed. Also, no interfacial damage was observed elsewhere along the SiC_w lengths. This lack of interface cracking or cavitation is indicative of strong bonding, which is necessary for the transfer of stress from matrix to whisker.

In order to further differentiate between the role of dislocations and that of SiC whiskers, a comparison was made between different samples of 2124 composite. One set of specimens was solution heat treated, quenched, and aged at 160°C to form precipitate phases. The material should then have an increased dislocation density associated with the precipitate formation, because of thermal expansion differences. Another set of specimens was solution heat treated, quenched, and not aged, but then tensile tested immediately. This second set of samples should have all alloying elements in solid solution, and thus there should

be no associated increase in density of dislocations.

The results of the tensile test comparison between the two systems are listed in Table II. Ultimate tensile strength, with and without particle strengthening, differs by only 7% , while the yield strength is found to increase by 20% when matrix precipitates are present. At low stresses, up to values sufficient for dislocations to cut through or bypass particles, these particles do contribute to the strength of the composite by acting as dislocation barriers. This result is expected from precipitation hardening theory [86]. However the very small difference in ultimate strength observed, indicates that once the applied stress exceeds the critical values for cutting or bypassing the particles, then the dominant strengthening mechanism is the transfer of load to the whiskers, which can support much higher stresses than the particle strengthened matrix alone.

Transverse vs. Longitudinal Strength:

Comparisons of ultimate strength and elongation-to-failure were made with the uniaxial stress applied in directions along the whisker axes (extrusion direction), and also with the stress axis normal to the extrusion direction. These directions are referred to as longitudinal and transverse, respectively. Longitudinal and transverse tensile tests were conducted on extruded, cold worked, and heat treated

Room Temperature Tensile Tests

Material	Ultimate Tensile Strength		Yield Strength	
	(MPa)	(Ksi)	(MPa)	(Ksi)
2124-T4	717	104	392	56.8
2124: 510°C 1 hr., CWQ (not annealed)	568	97.0	309	44.8

Table II.Comparison of strengths in 2124 composites, with and without matrix precipitates present.

materials. Comparisons of strength values and the fracture surface microstructures were then used to determine the effects of the thermal or mechanical processing. As seen by the results listed in Table III, transverse strength is approximately 75% of the longitudinal value in each instance.

Fracture surfaces in Figure 23 are from extruded 1100 matrix composites, showing similarity between transverse and and longitudinal fracture modes. The ductility exhibited by the matrix is quite evident. The same comparison for 2124 is shown in Figure 24. 2124 shows much more porosity and brittle appearing failure than 1100. This is attributable to the greater amount of brittle matrix precipitates present. An interesting phenomenon observed only in transverse specimens, and in fact, one which was seen in all transverse samples studied, is the large pieces of matrix and whisker conglomerate which are torn out in chunks, leaving behind large pores and exposed whiskers (see Figure 24-b). This result could be part of the reason for lower transverse strength, but the phenomenon is not fully understood.

Another fracture occurrance which was observed in most of the composites consists of whisker-free matrix that have been pulled to very fine knife-edges, while the surrounding areas retain the usual dimpled appearance. Such areas in four different alloys (1100, 2124, Al-Li-Cu-Mg, and Al-Cu-Mg) are pictured in Figure 25.

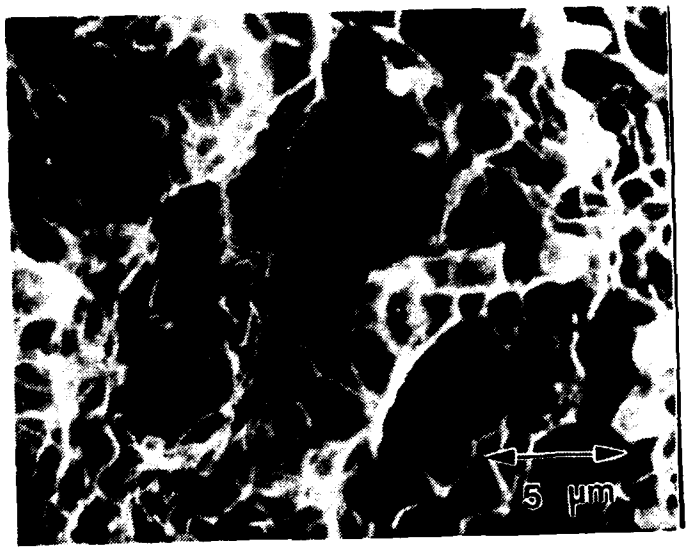
These transverse vs. longitudinal observations are

Table III

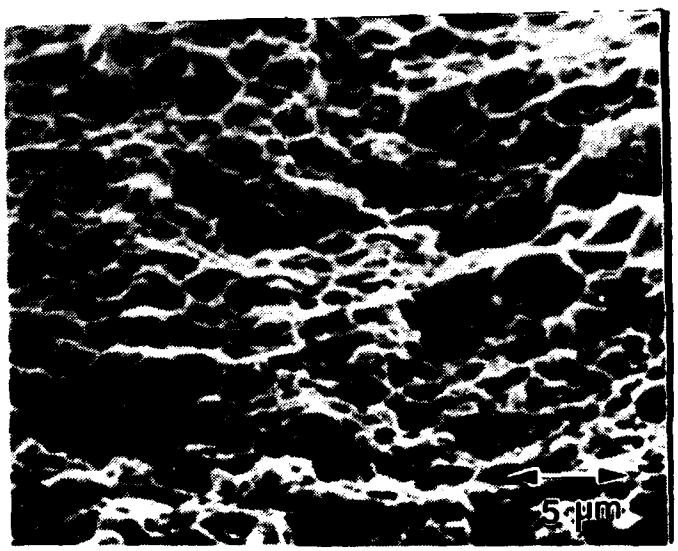
Transverse and Longitudinal Tensile Properties

Matrix	Orientation	Ultimate Strength (MPa)	(Ksi)	% Elongation
1100 (extruded)	L	89.9	57.9	4.9
	T	551	46.0	4.0
1100 (rolled) (52% reduction)	L	552	52.5	5.7
Al-Cu-Mg* (rolled)	L	396	57.5	8.5
	T	325	47.2	13.7
Al-Li-Cu-Mg* (rolled)	L	461	66.9	7.5
	T	518	75.1	8.4
2124 (extruded)	L	700	101.0	4.7
	T	469	72.0	4.3
2124 (hot rolled) (55% reduction)	L	520	75.0	5.5
	T	430	62.4	6.0
2124 (cold rolled) (18.7 % reduction)	L	441	64.0	7.8

* Size restrictions imposed by the rolled condition of this material limited the gauge length to less than 2.54 cm. Therefore, absolute values for % elongation are questionable.

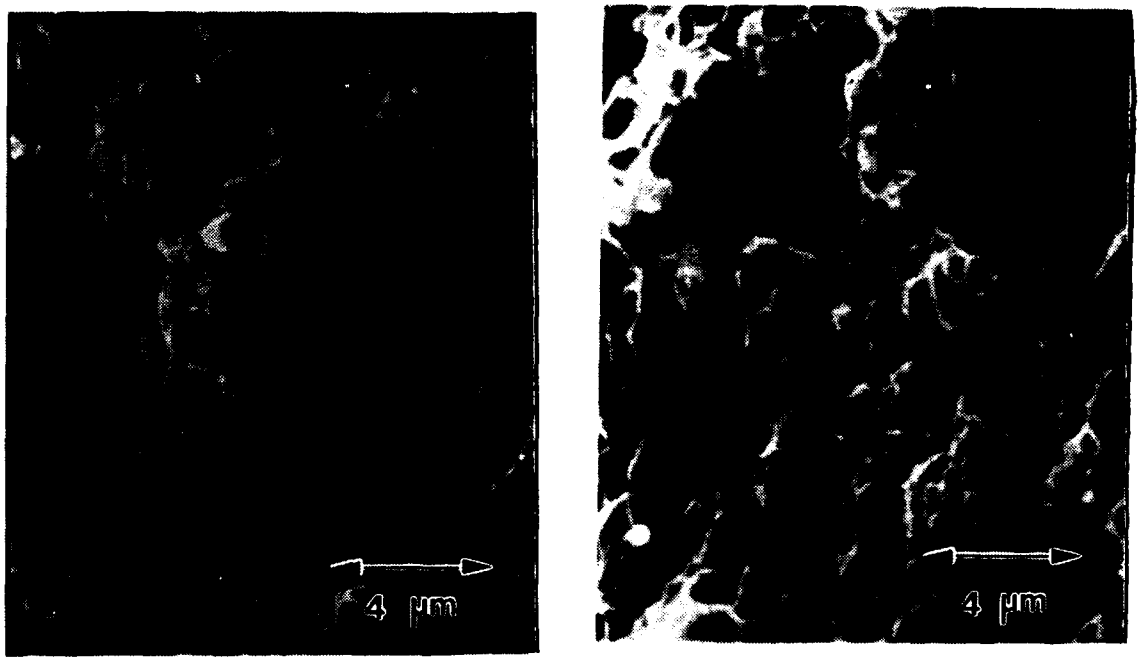


A

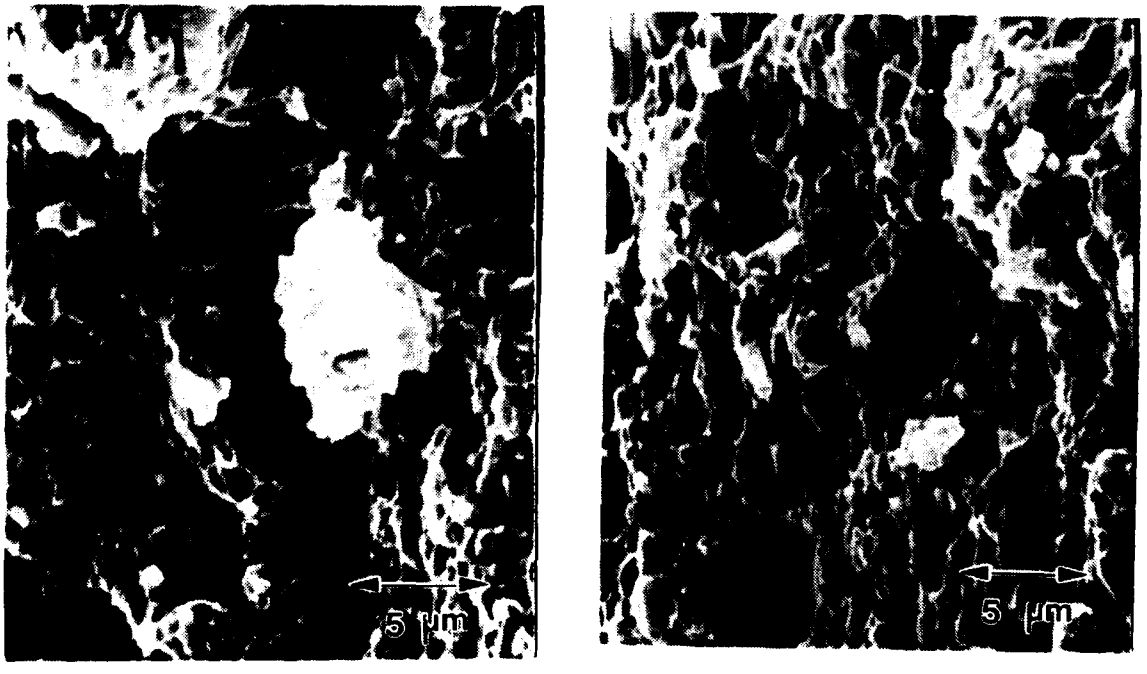


B

Figure 23. 1100 Composite fracture surfaces
(A) longitudinal (B) transverse



a



b

Figure 24. 7104 composite fracture.
(a) longitudinal (b) transverse

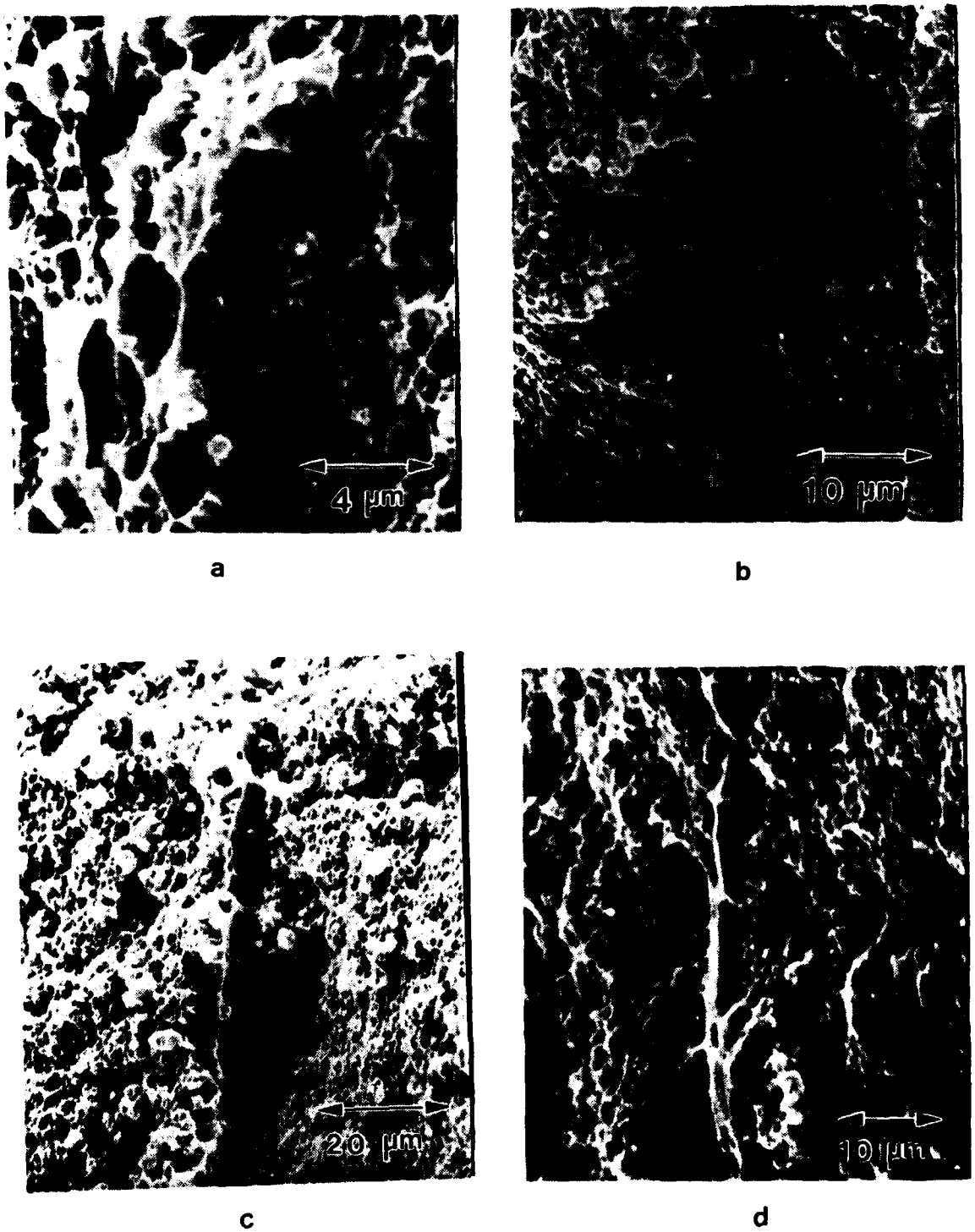


Figure 25. Whisker-free Matrix Ductility in (a) 2124 (b) Al-Li-Cu-Mg, (c) Al-Cu-Mg and, (d) 7100

consistent with the following analogy, as shown in Figure 26. When stress is applied to the whiskers longitudinally, the effective aspect ratio is greater than when the stress axis is transverse to the whisker length. In the latter instance, the "length" of the whisker is essentially the same as the diameter, and L/D = 1. Consequently, the smaller aspect ratio provides less strengthening in that direction.

Rolling Effects:

Transverse and longitudinal fracture surfaces of rolled Al-Cu-Mg and Al-Li-Cu-Mg are pictured in Figure 27. Whisker pullout, both the exposed whiskers and corresponding pores, can be seen in the longitudinal surfaces. These surfaces can be compared with those of material of higher strength in Figure 24. There the material is not rolled and interfacial damage (pullout) is not evident. In the rolled transverse fracture surfaces of Figure 27 and especially in Figure 27(C), the tearing away of material at the whisker-matrix interface is quite evident. Figure 27(D) also shows bundles of whiskers apparently not wetted by the matrix.

Data from 2124 transverse and longitudinal tensile tests are listed in Table III. Hot-rolling (55% reduction) decreased longitudinal tensile strength 25%, and cold-rolling (18% reduction) reduced strength 26%. In the transverse direction, hot-rolled tensile strength is 14% lower. It was verified, as has been seen at AMCO [30], that 2124 is not

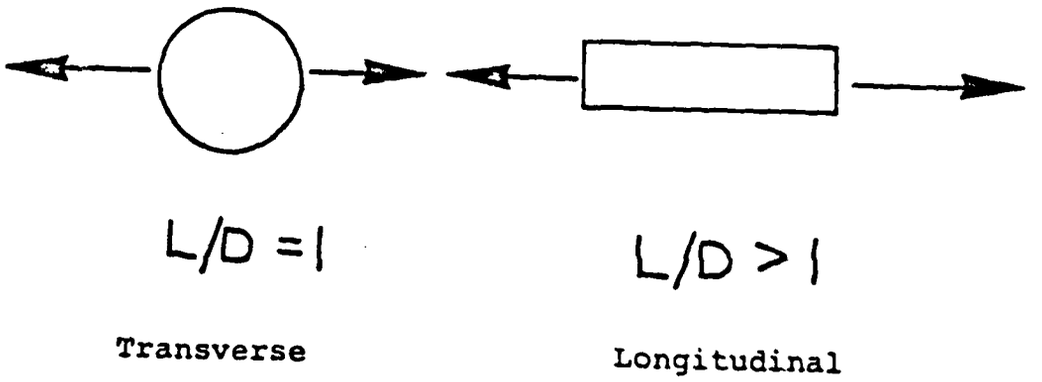


Figure 26. Comparison of Aspect Ratios in Transverse and Longitudinal Directions

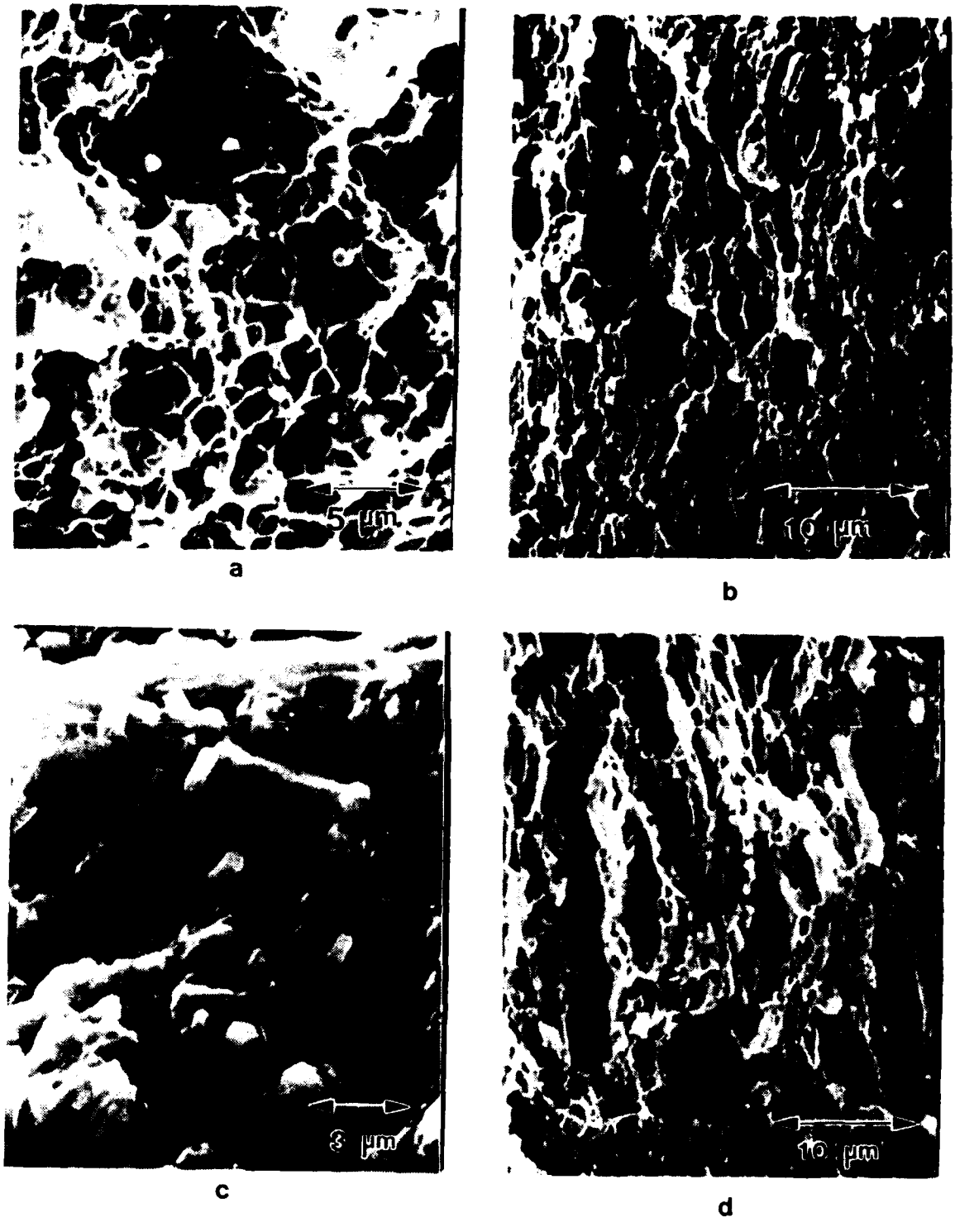


Figure VI. [illegible] by the Rolling Process: (a) [illegible] (b) [illegible] (c) [illegible] (d) [illegible]

not suitable for cold-rolling, since it fractures after only a few passes through the rolling mill. Hot-rolling at 496° C was found to introduce no visible damage, even after a 55% reduction in thickness.

Transverse and longitudinal fracture surfaces of hot-rolled 2124 are shown in Figure 28. These micrographs can be compared to Figure 24, which shows the same material in the unrolled state. Transverse fracture surfaces, before and after rolling appear similar. The longitudinal surfaces also have similar appearances, but there is an unusual observation in the hot rolled surface (Figure 28(a)). The area pictured, which was a common occurrance across the surface, does not exhibit the usual dimpled structure. Instead, areas of apparent localized incipient melting at a free surface (pores) are found. This melting phenomenon could have taken place during the hot-rolling process. The transverse surfaces again show the pulling away of fiber-matrix conglomerate, as seen previously. The fracture surfaces for 55% rolling reduction do not show any definite microstructural reasons for the observed strength decreases, but it is suspected that some interfacial damage must be incurred during the rolling process.

Differential Scanning Calorimetry and Heat Treatment:

The standard heat treatment for 2124 MC composites is a T6 designation, which consists of solution heat treating at

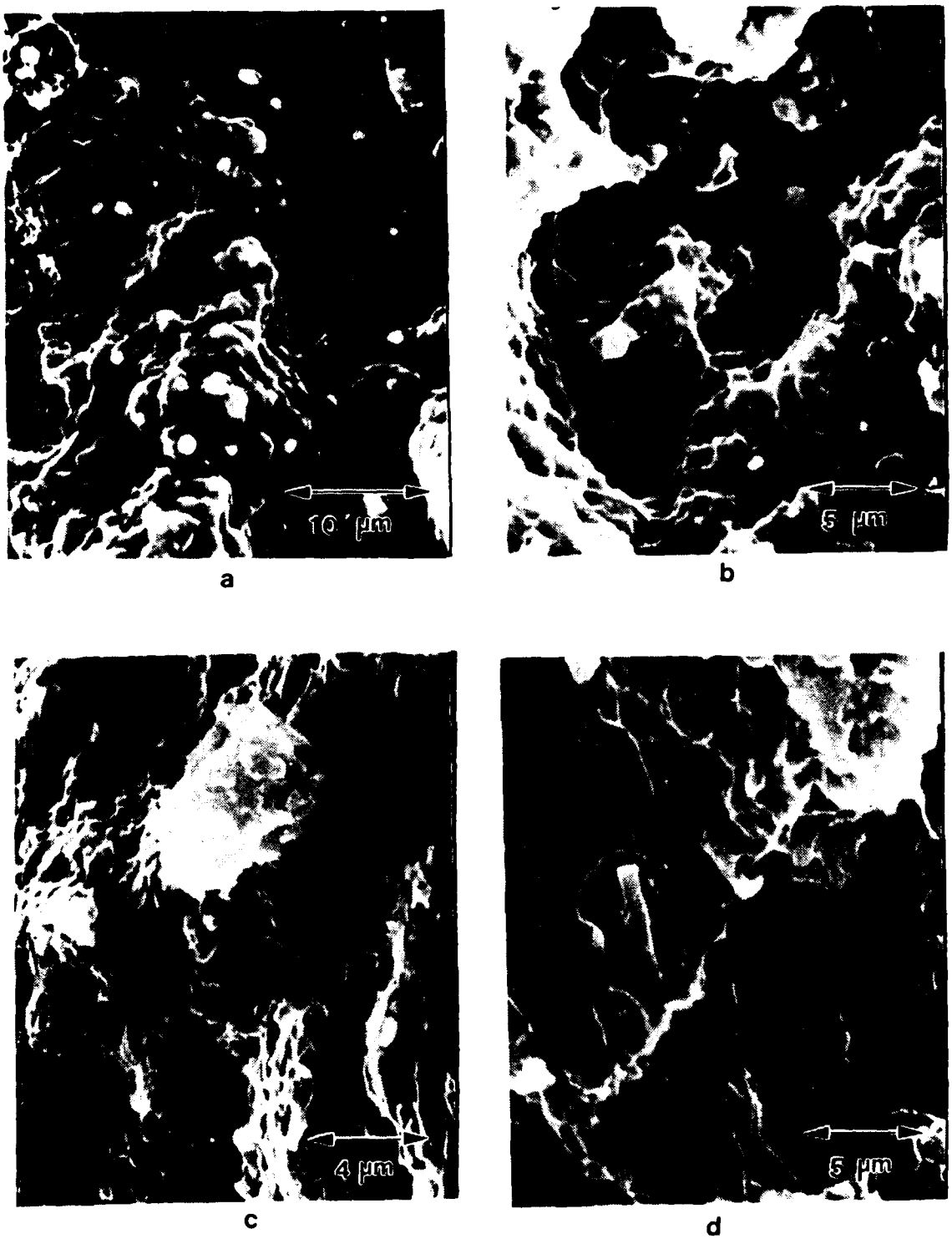


Figure 33. Fracture surface of 6121 Hot Rolled Composite (a) and (b) longitudinal (c) and (d) transverse

495° C, quenching in cold water, and then aging for 10 hours at 160° C. A study was done to investigate the effects of modifications of the solutionizing temperature.

DSC curves were generated for 2124 and 1100 composites, and are presented in Figures 29 and 30 respectively. Alloy 2124 is a precipitation hardenable alloy, while 1100 is commercially pure aluminum. The 1100 composite shows no apparent reactions at any temperature up to the melting temperature of 632°C. The absence of precipitation reactions is, of course, expected because this is not a precipitation hardenable alloy. Additionally, there is no visible interfacial reaction, or the energy involved in any such reaction is too small to be detected. For 2124, however, there are several temperatures of interest - 220°, 270°, 450°, and approximately 500°C. Instead of solutionizing at 495°, specimens were heated for one hour at these different reaction temperatures to allow each particular reaction more time to approach completion. Samples were then cold water quenched and aged, as usual. Three sets of rolled material (50%, 55%, and 60% reduction in thickness) and also sets of extruded 2124 specimens were heat treated at these temperatures. Control sets of 1100 composites (extruded and also rolled [50% reduction]) were prepared in an identical manner for comparison. Tensile tests were then performed on all specimens to correlate strength with heat treatment, and also to see if rolling deformation had any influence.

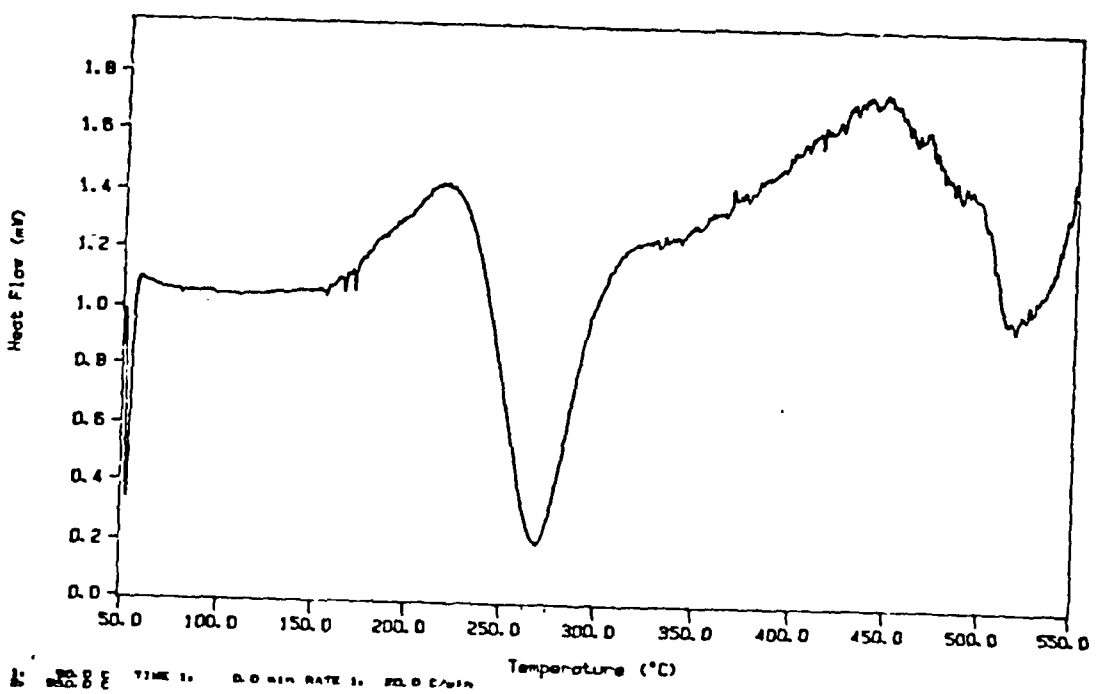


Figure 29. DSC Curve for 2124-SiCw

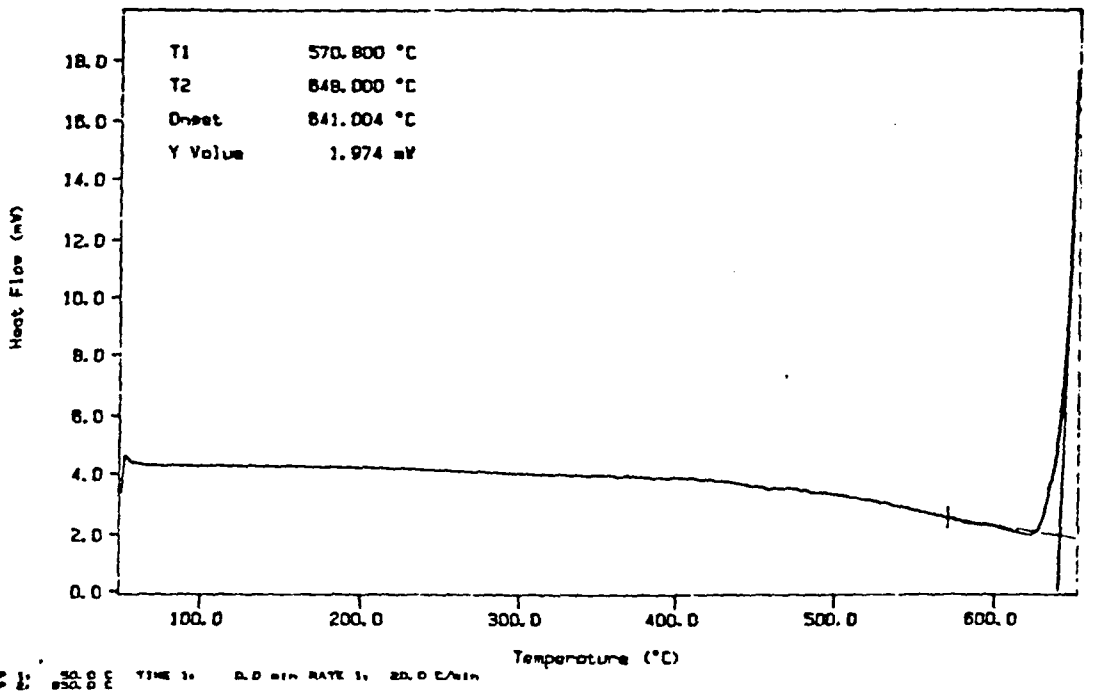


Figure 30. DSC Curve for 1100-SiC

A graph of ultimate tensile strength vs. heat treatment temperature is given in Figure 31 for the rolled and for the extruded 1100 materials. The extruded tensile strength is essentially unaffected by heat treatment, as would be expected from the generated DSC curve. The rolled 1100 shows an increase of strength as the heat treat temperature is increased. Additional points at 350° and 400° were generated to verify that there is a change in slope of the curve near 350°. Strength increases with temperature up to 350°, then plateaus off (see Figure 32). These results indicate that the interfacial damage induced by rolling, (as was previously described, see Figure 27), can be somewhat repaired by heating the material for 1 hour at 350°C or higher. This type of curve was only generated for material reduced in thickness by 52%. Greater thickness reduction would be expected to cause greater interfacial damage, which may be irrepairable. Fracture surfaces were examined, but no obvious differences were evident between any of the rolled or extruded materials.

The tensile strength vs. heat treatment curves for the various sets of 2124 composites are shown in Figure 33. The extruded material shows variation in strength with heat treatment - high strength after application of low temperature (220°), a decrease in strength after 270° exposure, and then again, high strength after T6 heat treatment. The rolled materials all exhibit curves of similar shape, showing relatively high original strength at 220°.

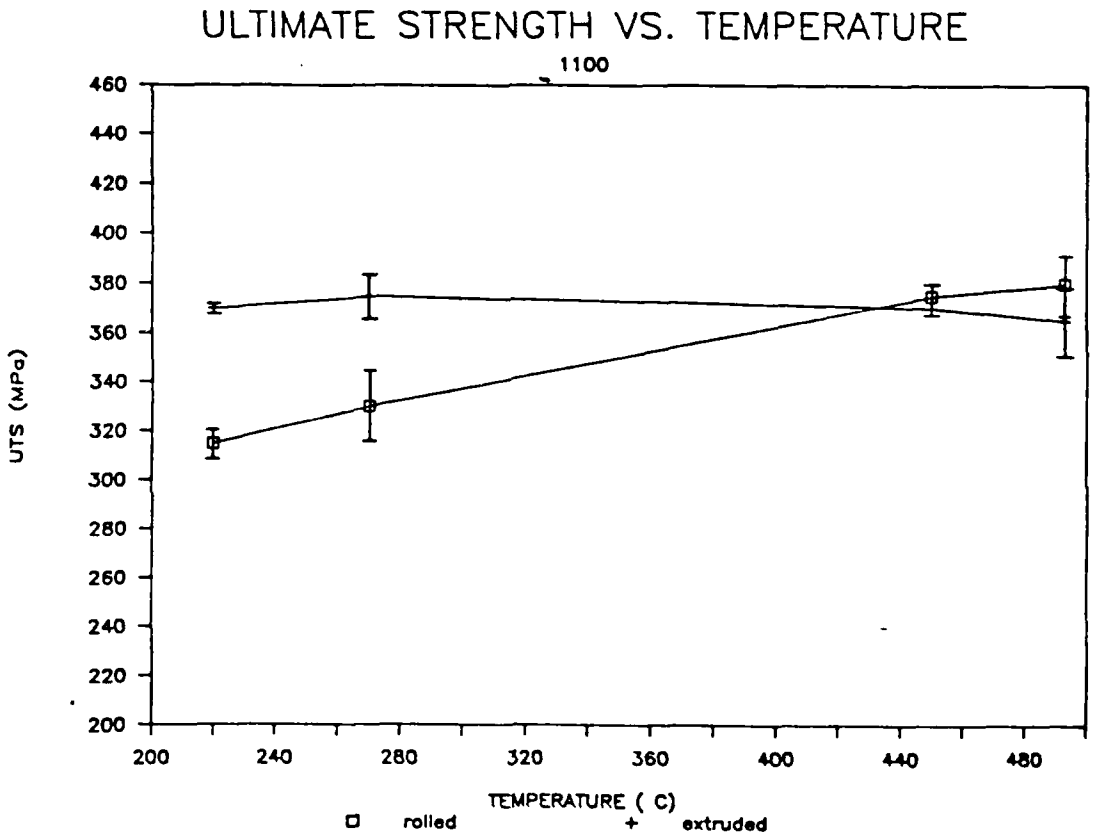


Figure 31. Tensile Strength vs. Heat Treatment Temperature for 1100 Rolled and Extruded Composites

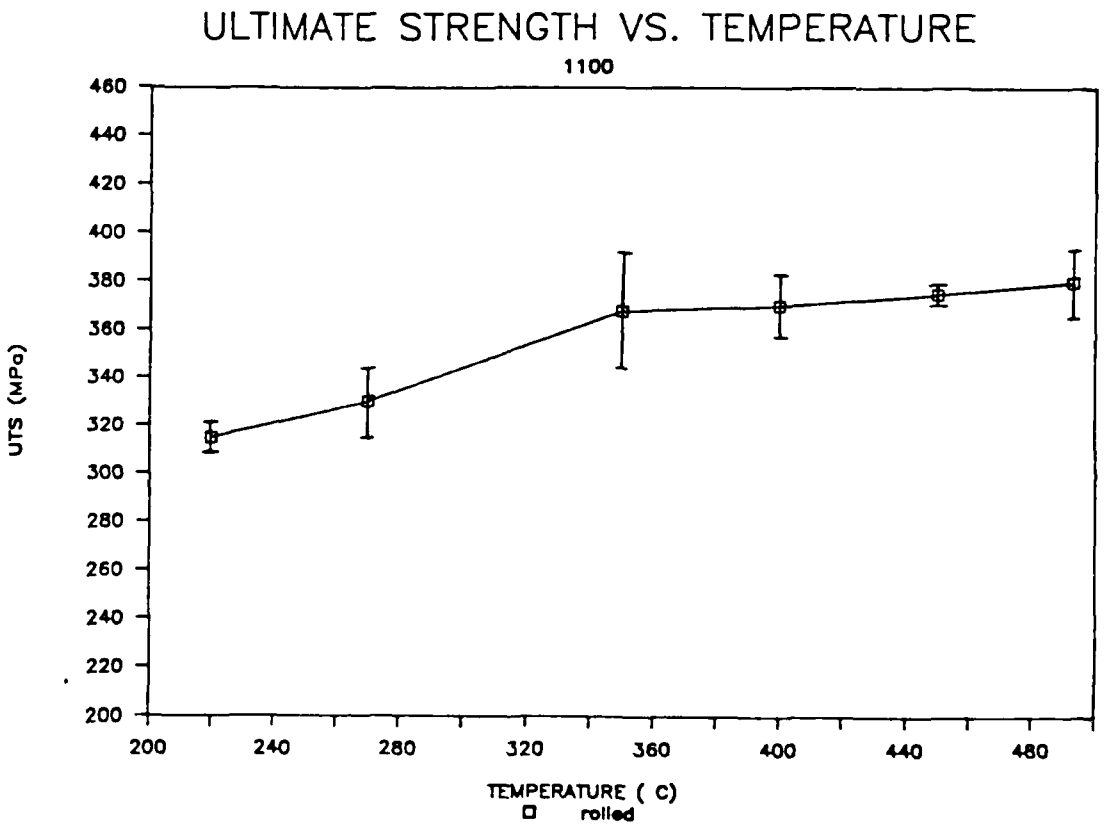


Figure 32. Tensile Strength vs. Heat Treatment Temperature For 1100 Rolled Composites

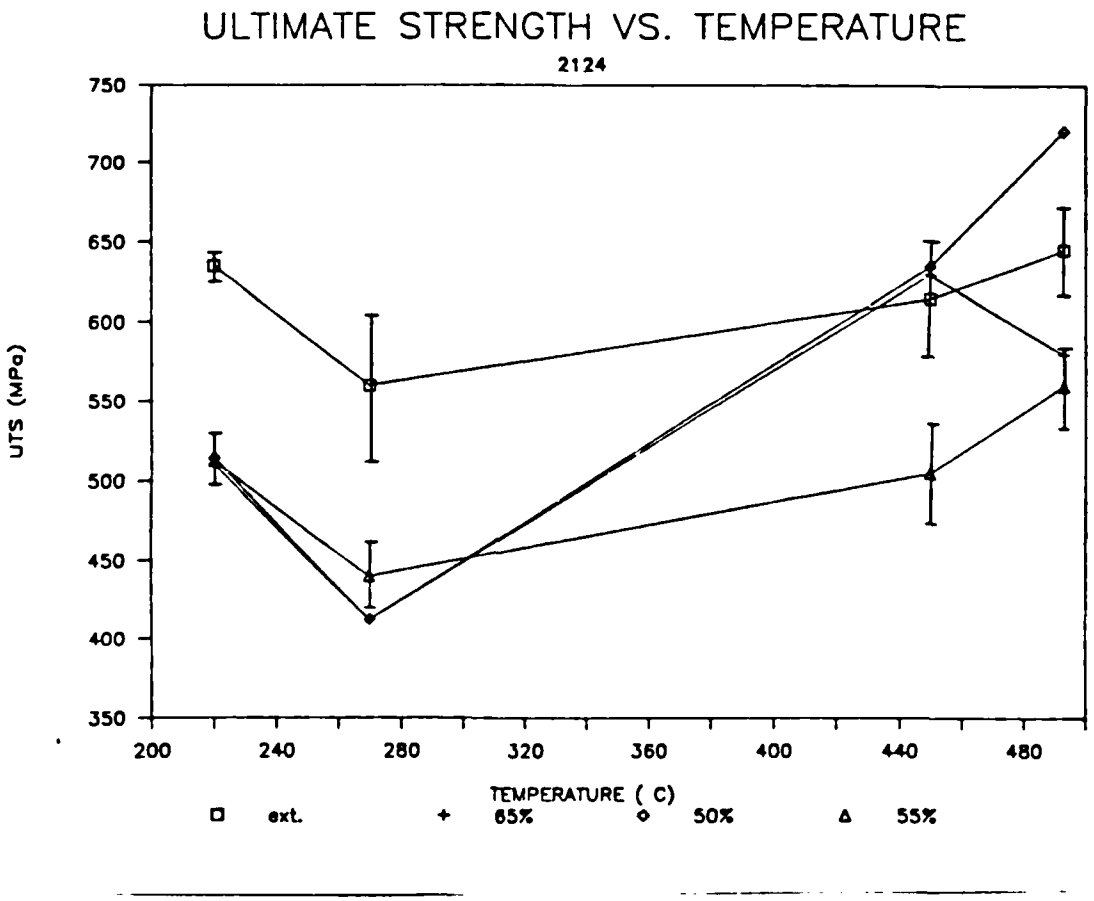


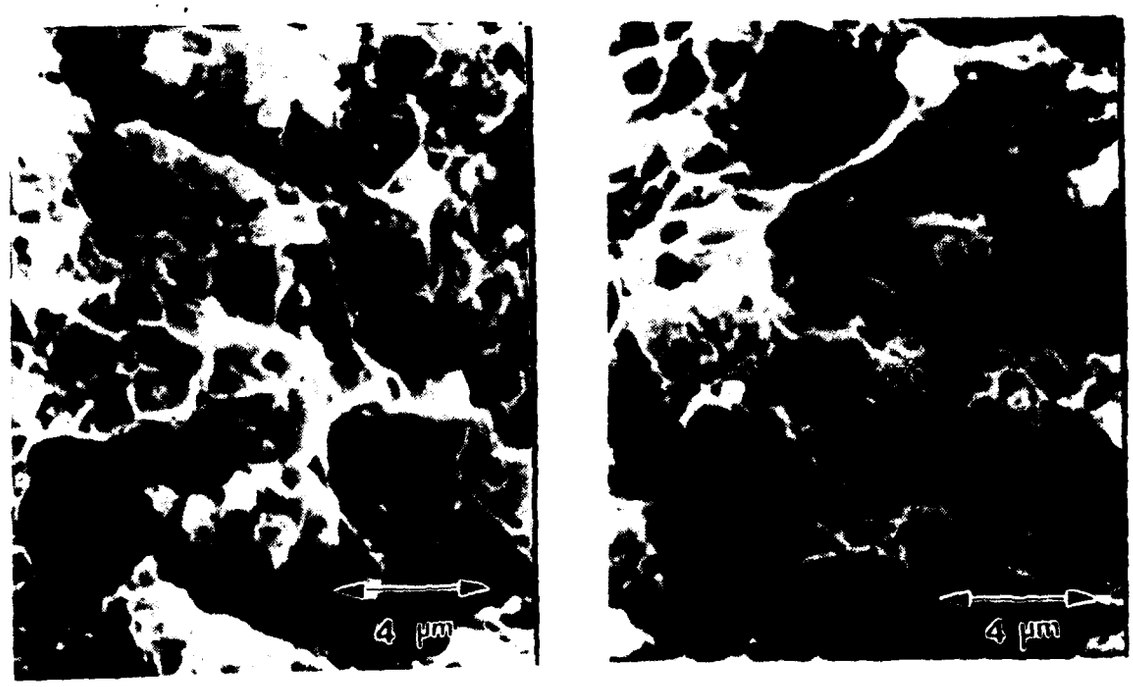
Figure 33. Tensile Strength vs. Heat Treatment Temperature For 2124 Rolled and Extruded Composites

then a large decrease in tensile strength at 270°, and the highest strength at 455°. (Recall Figure 6 in the background section, where the same type behavior was seen in 6061 composites.)

Reheating was seen to repair damage in 1100 composites. Although there are indications of a similar effect in 2124 near 500°C, there is scatter in the data for different roll reductions. Therefore, a definite conclusion can not be drawn.

The drastic reduction in tensile strength after only a one hour exposure at 270°C prompted further microstructural studies. T6 temper material fracture surfaces are shown in Figure 34, along with an energy dispersive x-ray spectrum from the surface. As expected, Al, Si, and Cu content is found due to the matrix, SiC whiskers, and Cu alloying precipitate particles (Al_2CuMg). (Mg content is detected at lower microscope accelerating voltages, but can not be seen in this particular graph.) The fracture surface exhibits the usual dimpled appearance of the matrix with precipitates and whiskers at the bottom of the dimples.

It is interesting to compare the T6 treated surface with the 270° treated surface pictured in Figure 35. Scattered areas of localized melting were found across the surface. Aside from the X-ray spectra differences, the melted areas on the 270° material have much higher Si content. An area of low Si content (top corner) has come a similar area



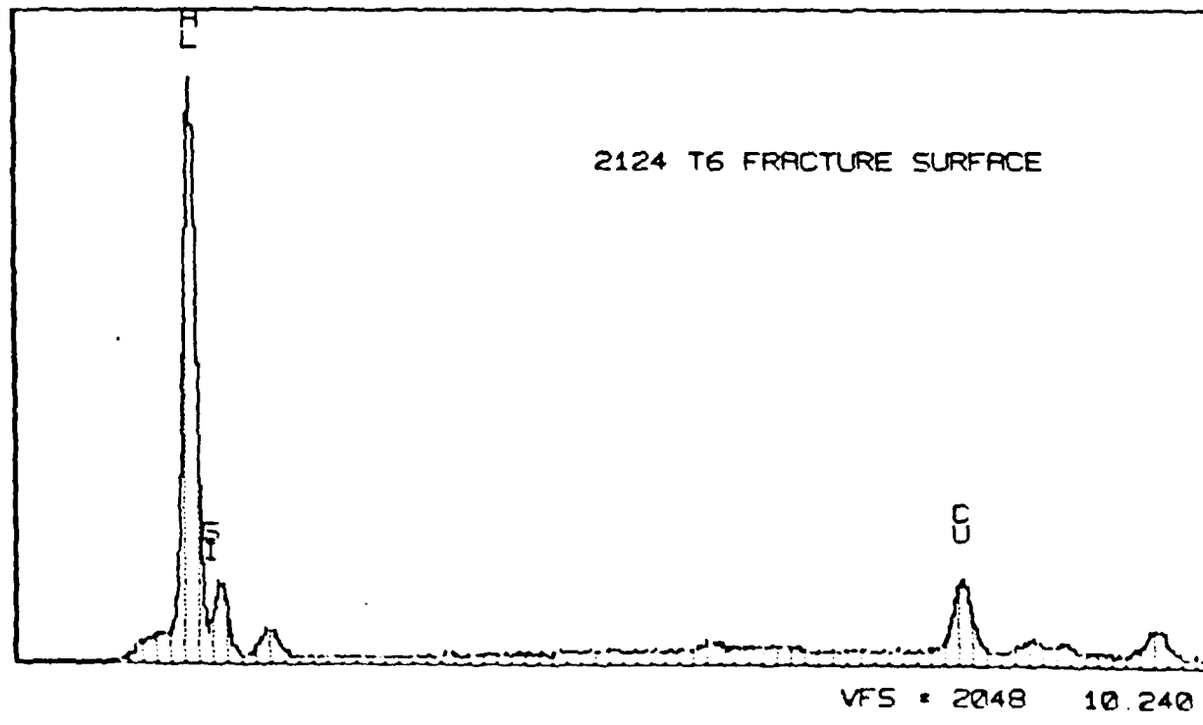


Figure 34. Fracture in 2124-T6 and Corresponding X-Ray Spectrum

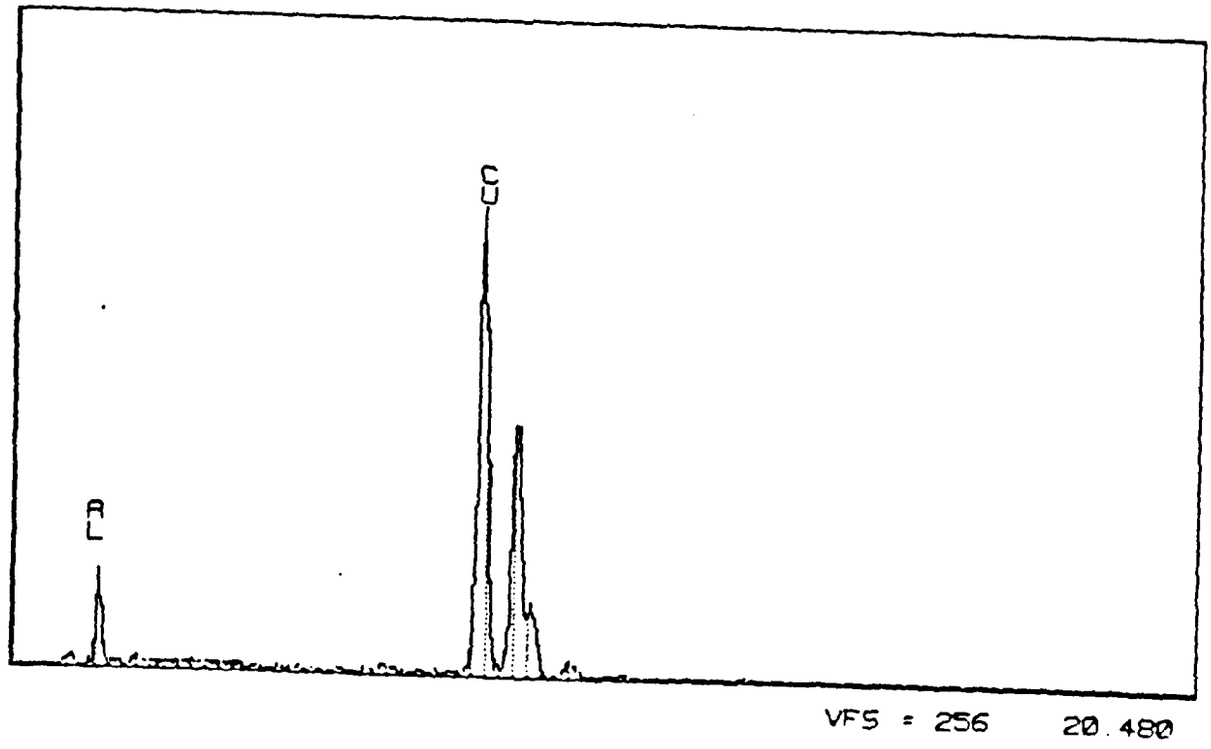


Figure 35. Fracture in 2124 Composite After 270°C Exposure and Corresponding X-ray Spectrum

of T6 surface. These Cu-rich areas of apparent localized melting are a probable cause of the poor strength properties of the 270°C treated material.

The TEM was used to verify precipitation of a Cu-containing phase and to further characterize its composition and morphology. In figure 40, micrographs show needle shaped precipitates within the matrix grains. Convergent beam energy dispersive x-ray spectra of the individual precipitates again indicate the Cu content of the particles. Fe is also found in these particles. Precipitates of similar size were not found in the T6 material (see figure 17).

The DSC curve (figure 24) indicates precipitation at 270°C. To verify that the reaction is due to matrix precipitation and not the SiC presence, another calorimetric curve was generated for a 2124 matrix with no SiC_w reinforcement. Composite and pure matrix curves are compared in figure 41. Note that the reaction at 270°C is of similar magnitude for both materials and appears to be independent of SiC presence. Since this reaction is a matrix phenomenon, TEM samples of 2124 alloy without SiC reinforcements were prepared from material treated at 270° for 1 hour. Micrographs (figure 42) show extensive, almost continuous precipitation along grain boundaries, and again the [illegible] precipitates. The [illegible] the grain boundary precipitates (figure 43) indicate a [illegible]

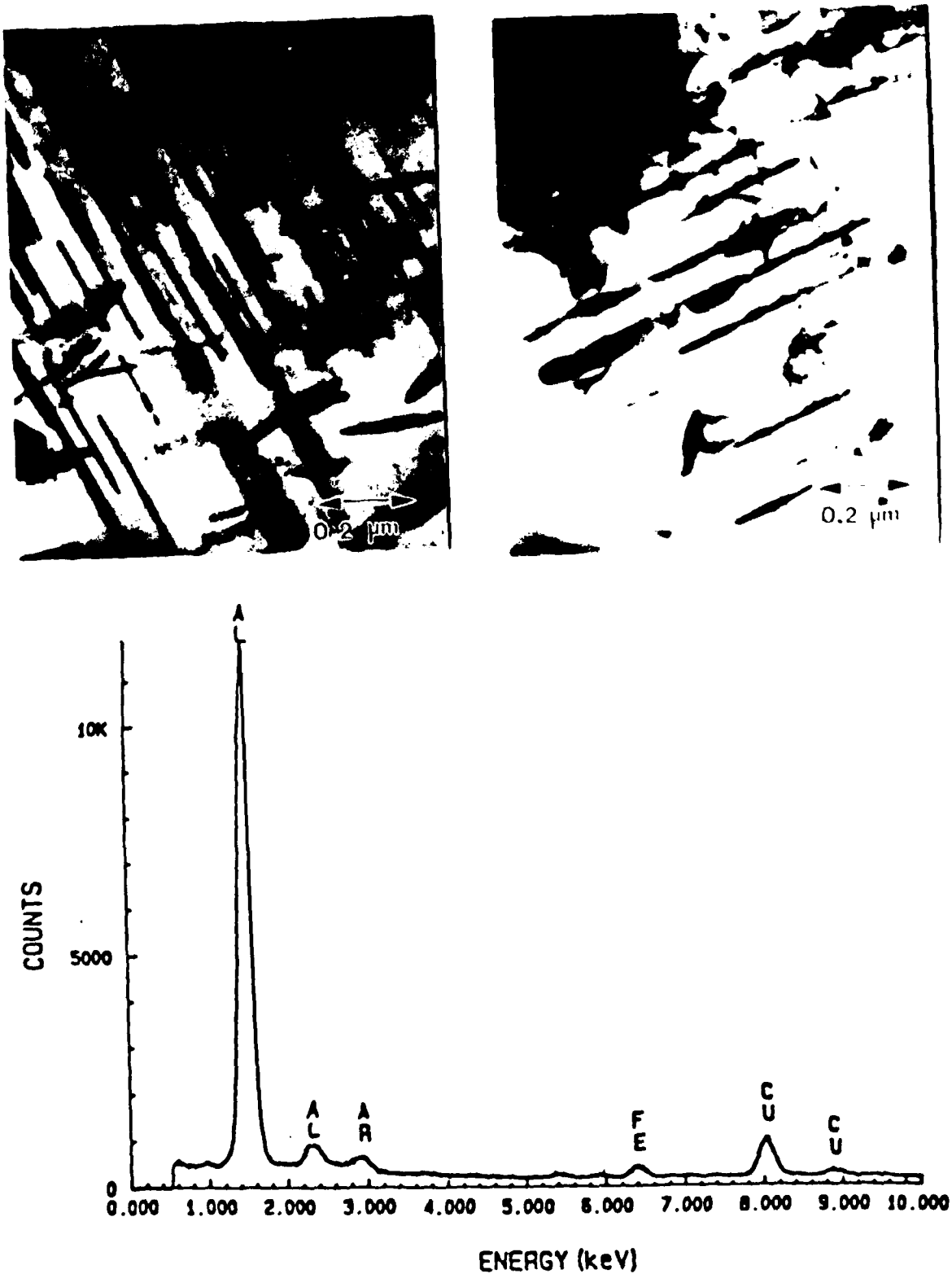
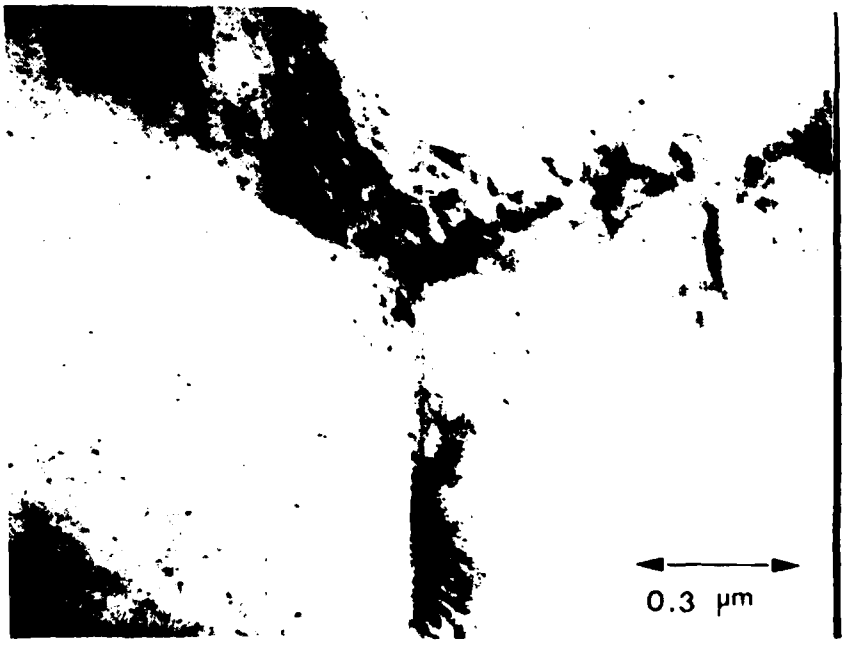


Figure 36. Needle-shaped Precipitates in Grains After 270° Exposure, and EDS of Al, Cu, Fe Content of Particles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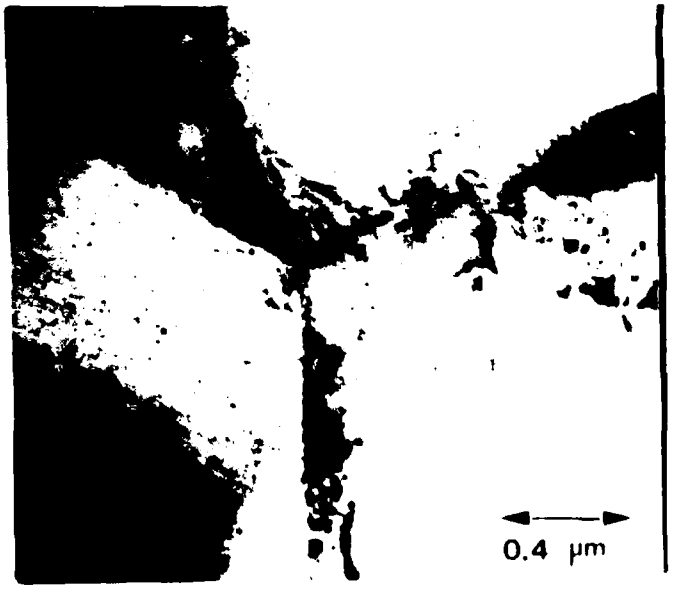
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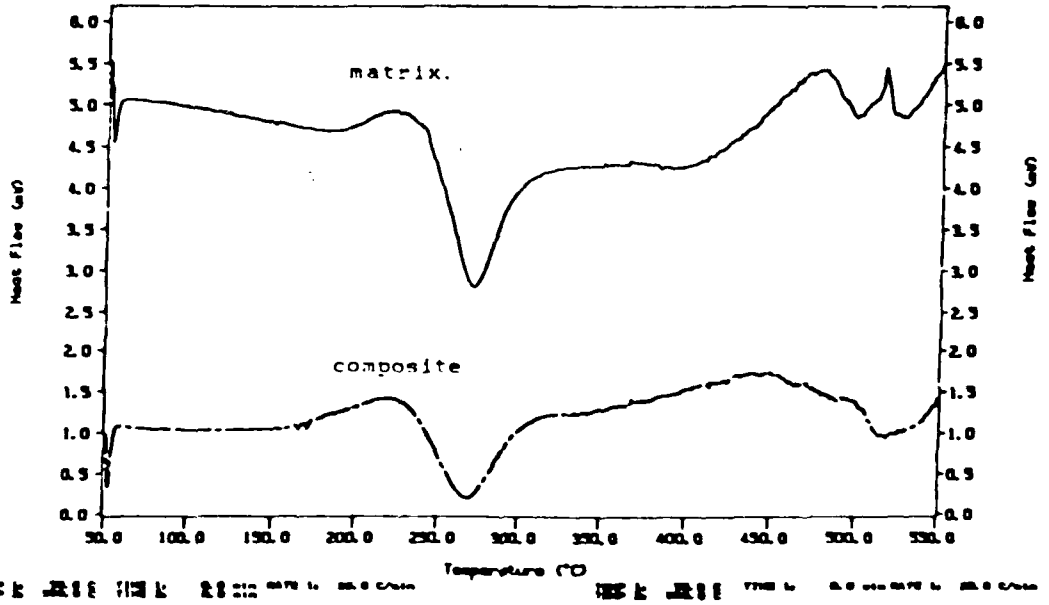
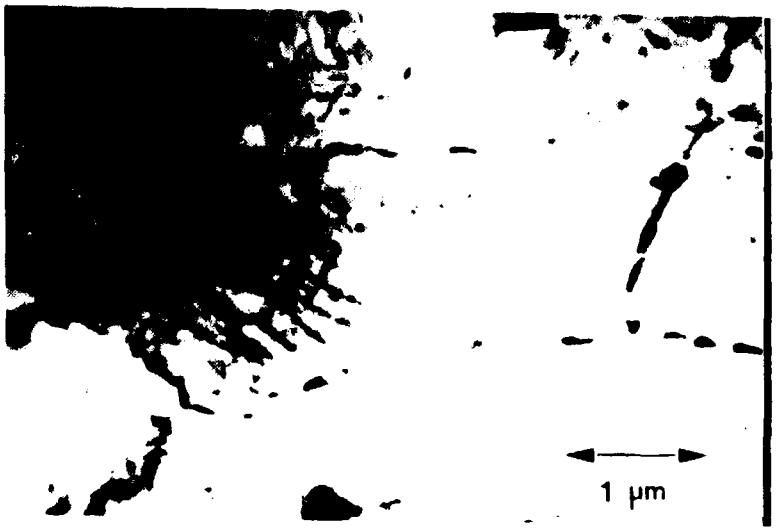


Figure 36. DSC Curve Comparison for 2124 Al, and 2124-SiC Composite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0.5 μm

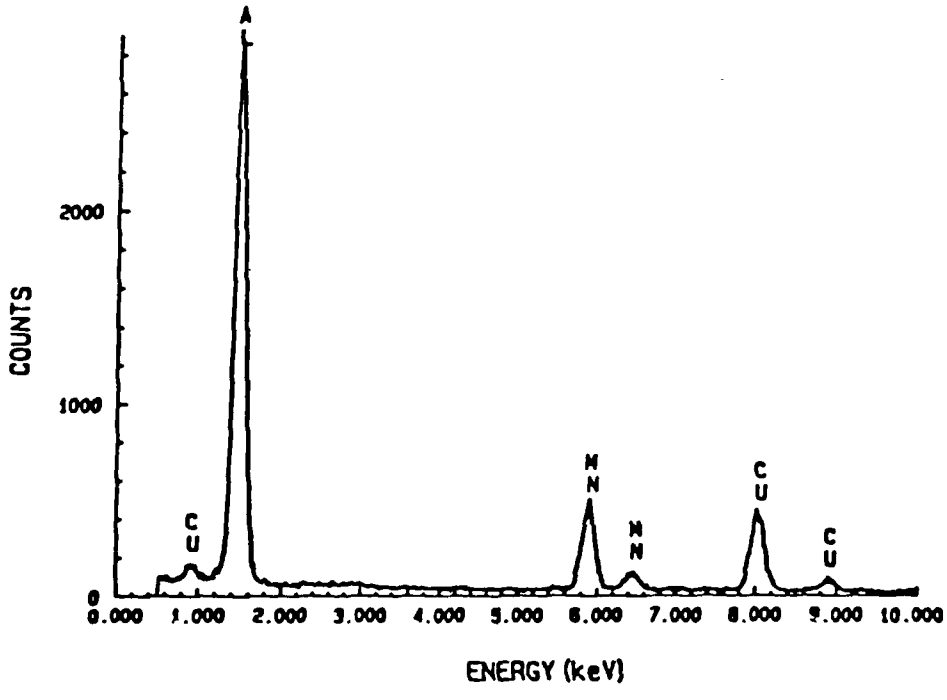


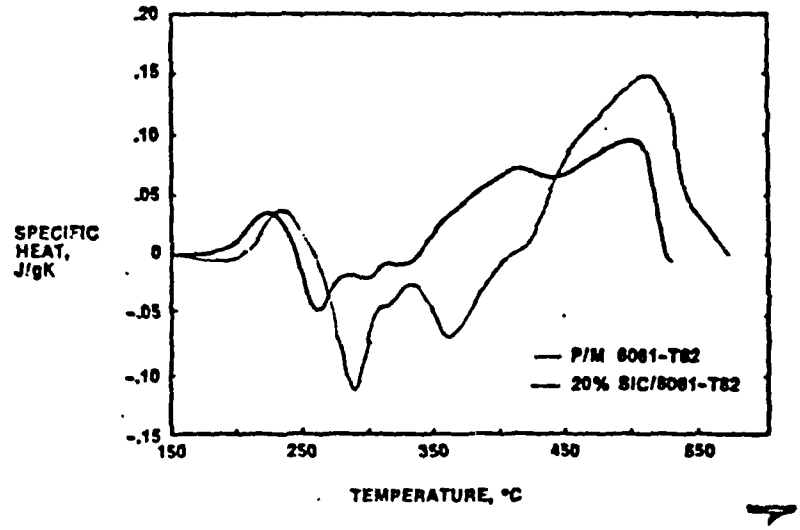
Figure 40. EDS Spectrum of Grain Boundary Particles in 2124

identified by electron diffraction methods, the usual form of Mn containing precipitates in this system is $Al_{20}Mn_3Cu_2$ [37], a brittle intermetallic. This precipitation is the obvious cause of the decrease in strength.

Figures 8 and 10 from the background section are reproduced in Figure 41 for comparative purposes. Recall that these two graphs were determined in different studies of 6061 composites by different investigators. The DSC curve shows a precipitation reaction at approximately 250°C in the composite. The tensile strength vs. temperature graph for the same type system, indicates lower tensile strength at this temperature. This effect in 6061 of lower tensile strength after exposure to a temperature where precipitation occurs, appears to be operating by the same mechanism as has just been demonstrated for 2124-SiC at 270°C.

Standard heat treatments for most aluminum composites are the same as those employed for the unreinforced matrix alloy. Temperatures and times of treatment are selected on the basis of reactions which occur in the aluminum alloy, such as the precipitation and growth of intermetallic particles. However, when SiC whiskers are introduced into a matrix, the reaction temperatures for the composite do not necessarily remain the same as for the pure matrix.

Figure 16 is a comparison of DSC curves for 2124 Al with no whisker reinforcement, and a 2124 matrix with 20 v/o SiC_w added. The curves are basically the same up to



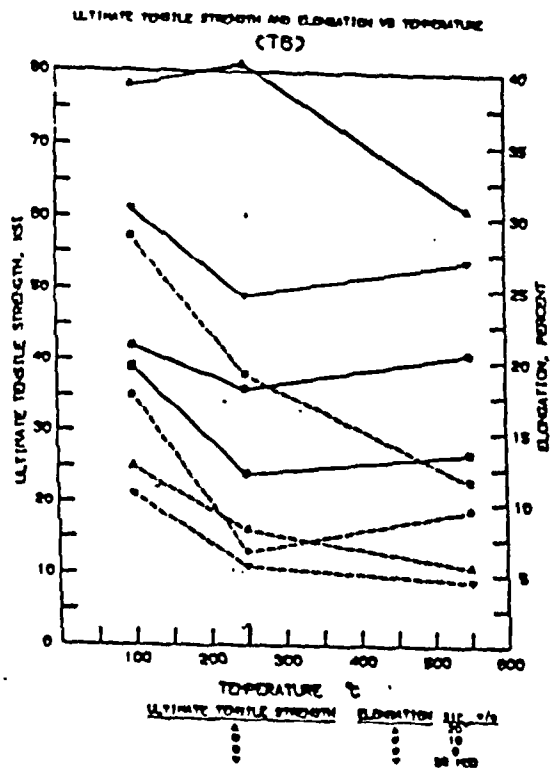
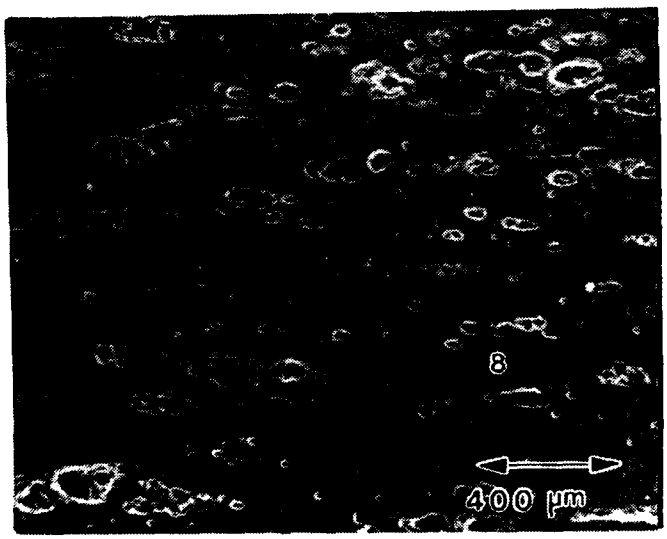


Figure 41. Tensile Strength and DSC Comparison in 6061-SiC System

approximately 475°C. At this point, the pure alloy exhibits an endothermic reaction. The sharp peak which begins just above 500°C corresponds to the solidus temperature of 502°C and the onset of melting. This same reaction at the same temperature does not occur in the 2124 composite. The reaction either does not occur at all, or there may be one or more reactions of opposite thermal energy which offset it, or cause it to occur at a different temperature. As stated above, the only difference between the two materials compared here is the presence of the SiC in the composite. It is possible that an interfacial reaction is taking place near 500°. Some of the energy which is normally attributed to the melting of the alloy, may now be contributing to a reaction at the interface instead. It is reasonable to attribute the thermal energy differences to the SiC_w presence. Figure 42 is a comparison of polished cross sections of pure 2124 matrix, and 2124-SiC_w composite, both of which were heat treated at 510° C for one hour, and then quenched. The pore size in the composite is much smaller, that is, melting is not as advanced as in the unreinforced material. It is not certain whether it is the SiC itself, or some other impurity, such as an oxide layer on the whiskers, which causes this phenomenon to occur. However, the difference at the solidus temperature must be considered when designing composite heat treatments.

a



b

Figure 49. Comparison of Degree of Melting in (a) 2124 Composite, and (b) 2124 Matrix, 510°C Heat Treatment

CONCLUSIONS

The integrity of the fiber-matrix interface was found to be a most important consideration in any processing of the composites studied. The strength and ductility of the material are both highly dependent on interfacial bonding. Unfortunately, the two properties are competitive; high strength requires a strong bond, yet the strong bond restricts overall ductility. Compositional and processing variations are necessary to find a balance between ductility and strength. The effects of some variations are discussed below.

Properties of Unprocessed Materials.

- Variations in fiber volume fraction and matrix composition have no major effect on aspect ratio.
- Discontinuous SiC whiskers with an average L/D ratio of 3 do carry a load in aluminum composites. The whiskers and interface are the major factors determining ultimate strength, while precipitation hardening contributes substantially to yield strength.
- Whisker alignment during the extrusion process results in anisotropic tensile properties. Ultimate strength transverse to the extrusion direction is 75-80% of the longitudinal value.

Mechanical Deformation:

- Rolling can produce dislocation strengthening in the matrix, but at the same time, it can also cause interfacial damage, resulting in lower strength.
- In some instances, (1100 matrix/20 v/o SiC_w), interface damage can be repaired by subsequent heat treatment.

Thermal Effects:

- Grain boundary precipitation in 2124-SiC_w composites, after exposure to 270°C, is quite detrimental to mechanical properties. Since the precipitate particles contain Mn, minimizing Mn content can possibly eliminate the problem.

  (A similar effect apparently occurs in the 6061 system, although it was not studied in this thesis. Any precipitation hardenable matrix may have temperatures at which unwanted segregation may take place. These phenomena should be identified, and either the temperature should be avoided, or the deleterious phase should be eliminated.)
- Thermal properties of Al matrices are affected by the presence of SiC whiskers. Alloying constituents can possibly react with the SiC. In 2124 composites, behavior at the solidus temperature (502°) is different from the unreinforced alloy. Melting is inhibited in the composite.

Recommendations for Fututre Work

Indications of interface repair by heat treatment should be investigated. Progress in this area could aid in the problem of difficulty in fabrication due to minimal ductility.

Various differences between Al matrices and composite materials have been shown. If better understood, these phenomena could result in better high temperature properties of the composites.

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APPENDICES

Appendix I

DAMAGE AND REPAIR OF THE INTERFACIAL REGION IN SILICON CARBIDE WHISKER REINFORCED ALUMINUM COMPOSITES

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INTRODUCTION

Discontinuous silicon carbide whiskers (SiC_w) can be added to aluminum alloy matrices to improve the strength properties of the material (1-3). To transfer stresses to the high modulus whiskers, an adequate interfacial bond must exist between the SiC_w and the surrounding matrix. In powder metallurgy composites this bonding can be achieved during vacuum hot-pressing at temperatures above the solidus. Materials are then hot extruded to align the whiskers.

This paper focuses on the breaking of bonds in this interfacial region during forming processes, and also indicates that these broken bonds can be reformed at relatively low temperatures.

EXPERIMENTAL PROCEDURE

Composite materials were obtained from Advanced [illegible] Materials Corporation in Greer, S.C. Three composites were studied - 2124 Al/ 20 v/o SiC_w, 1100 Al/ 20 v/o SiC_w, and a proprietary alloy, SXA24E +15v/o SiC_w.

Samples of the 1100 and 2124 composites were hot-rolled at 490°C to a 55% reduction in thickness (10% reduction per pass through the rollers) at the University of Virginia. Extruded specimens were rolled into 2 mm sheet at Advanced [illegible] Materials Corp. This sheet has an approximate [illegible] thickness of 80%.

Tensile tests were performed on both [illegible] extruded materials, using an Instron machine. [illegible] samples were tested at each condition. Ultimate [illegible] (UTS) values were obtained for extruded [illegible] measurements were then compared with [illegible] composites. The rolled materials were tested [illegible] as-rolled condition, prior to any solution [illegible] Rolled composites were also tested [illegible] consisting of a one hour exposure to 540°C [illegible] water quenching. No aging was performed.

Fracture surfaces were examined [illegible] scanning electron microscope (SEM). [illegible] and 1100 matrix composites were [illegible] extruded materials. Three [illegible] (TEM) disks were cut from [illegible] mechanically polished before [illegible] techniques. A Philips EM[illegible]

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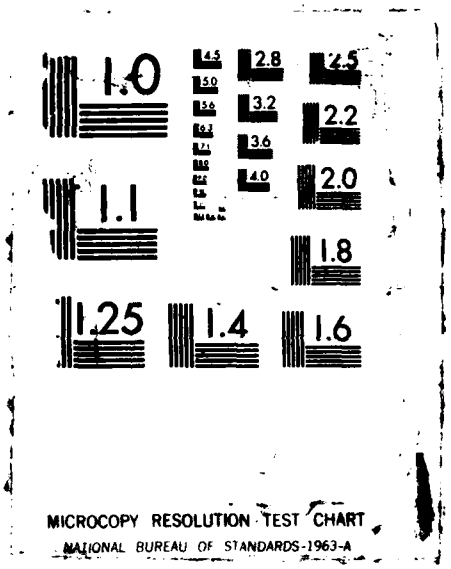
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used to examine specimens.

RESULTS AND DISCUSSION

Ultimate tensile strength data are presented in Table I. Values are given for extruded materials before rolling, for as-rolled samples, and also for rolled composites which were then heat treated. For each system investigated, UTS was greatly decreased after rolling. Upon subsequent heating at 495°C, however, the strength of rolled composites was found to be near the original values for the extruded composites. (Extruded SXA24E plate was not available for examination.)

	Ultimate Tensile Strength (MPa)		
Composite Material	Extruded	As-rolled	Rolled/Heated
1100-20v/o SiC_w	399	315	390
2124-20v/o SiC_w	700	430	673
SXA-15v/o SiC_w	*	356	668

* Extruded SXA24E+15v/o SiC_w was not available.

TABLE I. Ultimate tensile strength values for composites.

Yield strength values were not of primary concern in this study. It has been shown (4) that in SiC whisker reinforced composites, UTS is mainly controlled by the whiskers, while precipitate phases and whiskers both make substantial contributions to yield strength. As can be seen in Table I, UTS dramatically increases after reheating rolled materials. This increase is independent of precipitation hardening effects because the heating temperature of 495 °C is above the solutionizing temperature of both 2124 and SXA24E alloys. The heated materials were quenched and tested without being aged. UTS was chosen as the measurement parameter to isolate the whisker contribution, which is dependent on the strength of the matrix-fiber interface.

Examination of fracture surfaces indicate why strength decreases after rolling. Figure 1 is an SEM of the fracture surface of a high strength extruded sample of 1100-20 v/o SiC_w. In Figure 2, the surface of a rolled 1100 composite specimen, which has much lower strength, reveals a large amount of whisker pullout. The pulled out whiskers, along with the decrease in UTS, indicate that the fiber-matrix interface has been damaged during the rolling process.

Figure 3 is a schematic representing the cross section of a composite during the rolling process. One silicon carbide whisker, with an assumed circular cross section, is depicted

surrounded by aluminum matrix. The large arrows represent the external compressive forces exerted by the rollers. At the rolling temperature of 490°C, the matrix should be quite ductile and easily flow around the whisker. The small arrows indicate the probable stress state in the matrix near the whisker. Ashby (5) has proposed a dislocation model of plastic flow for this configuration, in which lateral tensile stresses at the interface can lead to separation and void formation. Since this rolling process is occurring at elevated temperature, vacancy motion and dislocation climb can occur, causing void formation. This is, of course, a very simple representation. Whisker interaction and misalignment, along with uneven distribution of the external forces greatly complicate the situation. However, it is reasonable to expect void formation at the interface between the brittle SiC and the ductile aluminum matrix.

TEM examination of extruded specimens of peak aged 2124 and also 1100 composites revealed no voids along the interface prior to rolling. Figure 4 shows, however, that these voids do indeed occur in rolled materials. In the figure, the dark regions are the whiskers (in cross section), and the voids are clearly shown as the light semi-circular areas adjacent to the fibers.

An in-situ heating experiment was done in the TEM. In Figure 4(a) an interfacial void is shown in a sample of rolled 1100 composite before heating. The series of micrographs in Figures 4(b) through 4(e) show the same void as time progresses and heat is applied. The photos indicate that the void is being eliminated during heating. The last micrograph, Figure 4(f), shows that the void remains sealed after cooling to room temperature.

Care must be taken when comparing thin-film heating and diffusional processes to similar processes in bulk material. Therefore, the time duration and temperature during this in-situ experiment was not accepted as absolute. Rather a separate temperature study was undertaken. Figure 5 shows a plot of UTS vs. one hour heat treatment temperature for rolled 1100-SiC composite material. This graph indicates that increases in strength occur not only near 500°C, but also at relatively low temperatures. Heating for one hour at 350°C results in nearly the same ultimate strength as that obtained after heating at 495°C. However, the exact temperture at which interfacial repair begins has not been determined.

The exact nature of what has been referred to as interfacial repair is not known. Damage may be occurring in the matrix material immediately adjacent to the whisker-matrix interface rather than actually breaking the interfacial bonds themselves. The actual structure and composition of interfacial bonds in SiC_w composites has been the subject of much debate. Depending on the matrix composition, various compounds such as Al_2O_3, Al_4SiC_4 (6), and MgO (7) have been reported as occurring at the interface. Certainly, different interfacial structures in different composites would not be expected to have the same thermal

characteristics. The above results, however, show that this damage and repair effect occurs in composites of varying compositions. To determine exactly what processes are occurrring and where they take place will require analysis on a much finer scale than that used in this study.

CONCLUSION

Bonding between the matrix and reinforcement in silicon carbide whisker/aluminum matrix composites plays a major role in the strength of the composite material. Forming processes such as rolling can cause damage to the interface. This damage can be alleviated by subsequent heat treatment at relatively low temperatures.

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Figure 1. Fracture surface of a high strength extruded sample of 1100/20 v/o SiC_w composite

Figure 2. Fracture surface of a rolled 1100/20 v/o SiC_w composite

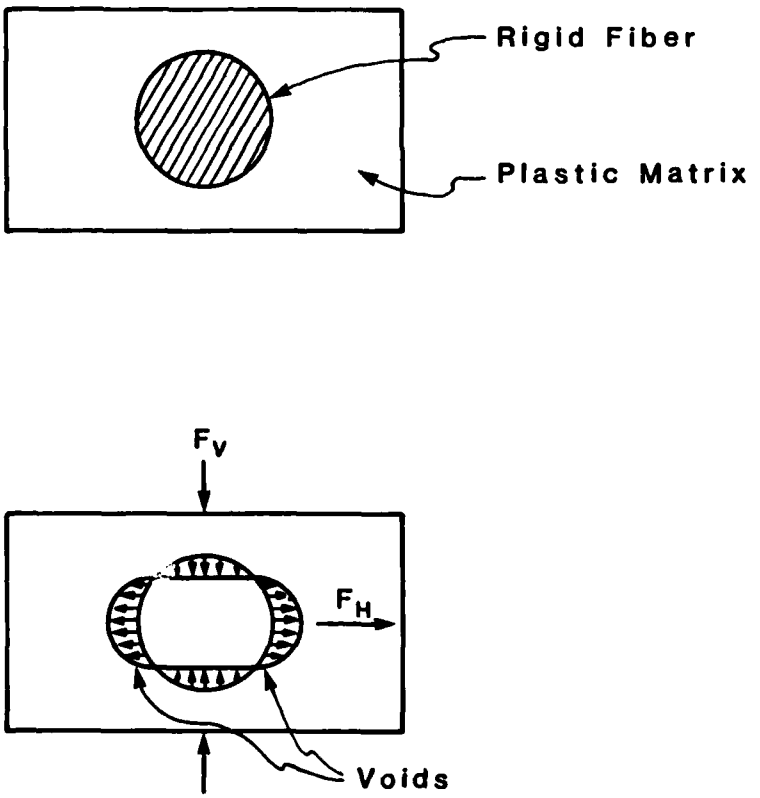


Figure 3. Schematic representing forces during rolling

Figure 4. TEM of void at interface caused by rolling

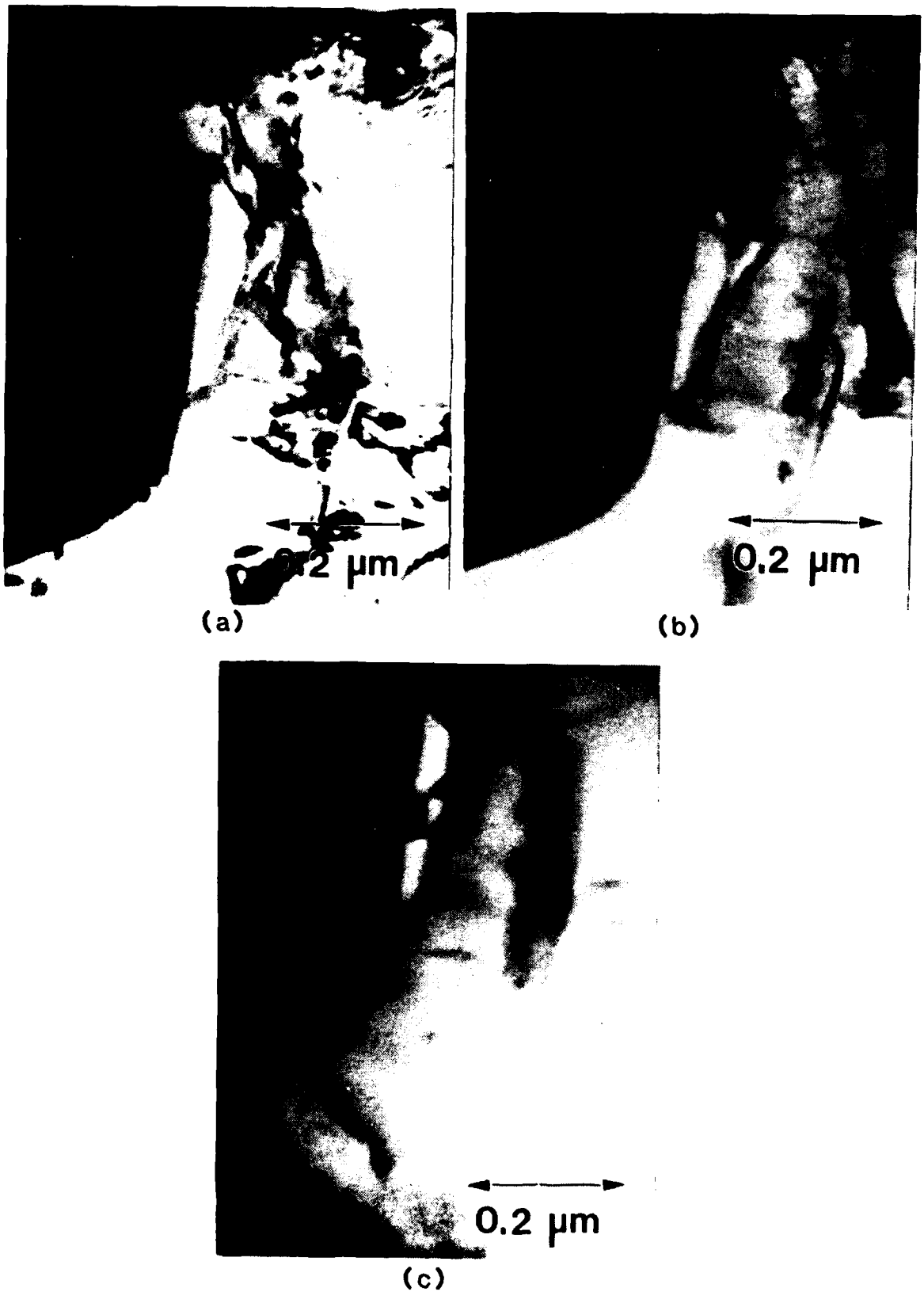
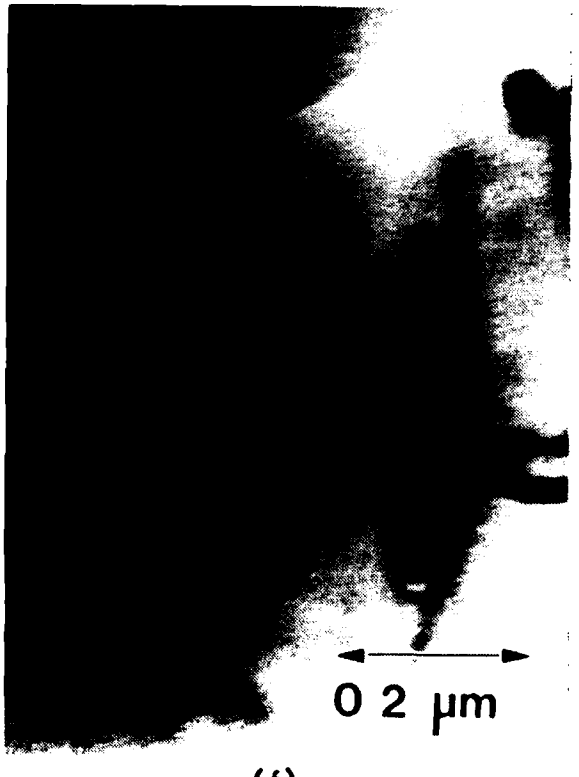


Figure 4. a-f Showing closure of void with time and temperature in in-situ heating experiment

(d)

(e)



(f)

Figure a-f cont'd

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